ENGINEERING PROPERTIES OF MINIATURE CEMENT - FLY ASH COMPACTS PREPARED BY HIGH PRESSURE COMPACTION

A Thesis

by

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ABSTRACT

Engineering Properties of Miniature Cement - Fly Ash Compacts Prepared By High Pressure Compaction (August 1985)

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Current practices and theory conform to the concept that for specialized uses a portland cement concrete material with strength approaching 20,000 psi may be produced by reducing the amount of porosity present in the microstructure of the paste-matrix material. To accomplish this reduction in porosity, high pressure compaction and increased formation of cementitious products from a pozzolanic reaction have provided excellent results individually. However, judging from an extensive literature survey, the combination of the two has not been examined.

The purpose of this investigation was to determine the combined effects of fly ash as a partial replacement for portland cement and the use of high pressure compaction in reducing the porosity and increasing the compressive strength of prepared miniature compacts of approximately one cubic centimeter in size. The three parameters investigated included the initial porosity level, the percentage fly ash replacement of the cement and curing duration.

The results of the study revealed that high pressure compaction is an excellent means of producing a low porosity condition and ultimately a greater strength in a cementitious material. Confirming earlier findings, it was found that increased hydration product

formation occurs under conditions of greater porosity. Concerning the combination of cement and fly ash under high pressure compaction, it was found that particle gradation and compatibility are important considerations when producing the compacts. The existence of an optimum percentage of fly ash replacement for cement in the prepared compacts could not be determined. The expected increase in strength due to the pozzolanic reaction was not apparent as increasing amounts of fly ash provided for decreasing strength levels. The reason for this is believed due in part to inadequate curing duration allowing for incomplete formation of the pozzolanic reaction and also to the amount of necessary calcium hydroxide available in the low porosity compacts.

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CHAPTER I

INTRODUCTION

General

With current concrete technology, the production of concrete with compressive strengths from 10,000 to 12,000 psi has become quite common. Generally, the attaining of strengths at this level involves the coupling of the optimization of the constituent materials, namely cement, water, aggregate and special additives, with excellent quality control procedures. Current practices and theory conform to the concept, however, that for specialized uses a portland cement concrete material with strength approaching 20,000 psi may be produced by reducing the amount of porosity and strength-reducing void space present in the microstructure of the paste-matrix material. To accomplish this goal, the utilization of high pressure compaction and increased cementitious binding due to the addition of pozzolanic admixtures such as fly ash provide excellent possibilities.

Study Objectives

The objective of this study was to determine the combined effects of fly ash as a partial replacement for portland cement and the use of high pressure compaction on the compressive strength of prepared

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Note: This thesis follows the style used in the Journal of the American Concrete Institute, published by the American Concrete Institute.

miniature compacts of approximately one cubic centimeter in size for various durations of moist curing. Also of concern were the effects of high pressure compaction on the porosity, the hydration process and the pozzolanic reaction in the prepared compacts. An additional objective was to produce data which could be utilized in a separate investigation involving the production of ultra-high strength mortar and concrete cubes produced by high pressure compaction on a much larger scale.

Study Scope

This study consisted of extensive research into the theory and principles involved in the production of an ultra-high strength cementitious material through high pressure compaction and pozzolanic reactions, a determination of the present state-of-the-art regarding their use in concrete technology, and a laboratory investigation of certain test parameters to determine their effect on producing an ultra-high strength cementitious material.

The test parameters chosen for study in the laboratory investigation included: 1) percentage porosity, 2) percentage of replacement of portland cement with fly ash, and 3) curing time of the prepared compacts.

CHAPTER II

LITERATURE REVIEW

Present Status

In recent decades, the utilization of concrete which develops compressive strengths of 10,000 to 12,000 psi in relatively short times has experienced increased use in the construction industry.[1,2] The production and usage of concrete with strengths significantly greater than this remains, however, more difficult to achieve on a reproducible basis. This is due mainly to the many different variables which influence the development of strength in a heterogeneous material such as portland cement concrete.[3] In practice, for the production of concrete at the 10,000 to 12,000 psi strength level, the state-of-the-art has advanced to the stage where it is generally a matter of optimization of the constituent materials, aggregate, cement, water, and various admixtures in the proper combinations, and the utilization of excellent quality control in the mixing, batching and placement techniques.[4,5,6,7,8]

To produce a concrete with compressive strength at the ultra-high strength level of 20,000 psi, in addition to the optimization of these strength influencing factors, it is necessary to significantly increase the strength of the weaker binder or matrix material in a given concrete batch. Current practices and theory conform to the concept that the amount of porosity present in a matrix material is the most influential factor concerning strength and that to produce an

ultra-high strength material the microstructure of the matrix must be modified in such a way that the porosity level is significantly reduced.[3,9,10,11,12,13] To accomplish this goal, the utilization of high pressure compaction and increased cementitious binding due to the addition of pozzolanic admixtures such as fly ash provide excellent possibilities.

The Nature of Strength in a Cementitious Material

A complete discussion of the nature of strength in a portland cement concrete or cementitious material is very complex and beyond the scope of this paper. Of importance are the major items which have been found by previous research to have the greatest effect on the strength of a portland cement material.

The strength in such a material is dependent upon 1) the strength of the aggregate material, 2) the strength of the matrix material, and 3) the strength of the bond between the matrix and aggregate material. In order to understand the concept of strength, of primary concern is the most basic ingredient, the paste or matrix material which consists of hydrated, partially hydrated and unhydrated cement material in the presence of water. The matrix is actually the "glue" which binds the various sizes and types of aggregate together in a concrete batch. Since quite often the aggregate used has far superior strength than that of the matrix material, it is the strength of the matrix material which must be improved for a higher strength material to be achieved.[3]

The microstructure of the matrix material is the controlling

factor for matrix strength.[3] The matrix is initially a two component system consisting of anhydrous cement and water. Upon exposure of the cement particles to water, the major cement compounds, namely the tricalcium silicate (C_3S), the dicalcium silicate (C_2S), the tricalcium aluminate (C_3A) and the tetracalcium aluminoferrite (C_4AF) begin the hydration process and the formation of the two major hydration products, cement gel and calcium hydroxide, begins. The majority of the cement gel is composed of calcium-silicate-hydrates (C-S-H) which are primarily responsible for strength development in a matrix material. The calcium hydroxide is believed to provide no major impact on strength other than the occupation of detrimental void space and increased surface area contact between the different particles of the matrix.[13] Calcium hydroxide is essentially a by-product of the hydration process which forms only in available free space.[13] As presented later, this by-product becomes an essential part of the hydration process in the presence of a pozzolan.

As the hydration process proceeds, the gel product formed effectively coats the cement particles with various sized and shaped layers of hydration products. At a certain time after the beginning of the hydration process, the matrix material consists of the cement gel, remaining unhydrated cement material, available free water and void space or porosity. The majority of the porosity has developed as the hydration of the cement particles has "used" the available water in the vicinity creating a remnant of water-filled space in the hydrating paste. The amount of cement gel or hydration product will continue to increase while there is sufficient water available in the

vicinity to encourage the hydration process. Current theory states that the cement gel product can only form in the void space provided by the exchange of available free water through the hydration process. During this process, the surface area of the gel solid product increases tremendously.[3,12,13,14]

The strength of the cement paste is developed as the cement gel hydration product continues to form around each cement particle and expands outwardly as the hydration process proceeds, increasing the amount of surface area contact between the various components in the paste. It is generally believed that two types of cohesive bonds give the matrix material its strength. One is the physical attraction between the surfaces of the hydrated cement particles generally characterized as van der Waals forces. The second type of cohesion pertains to the interlocking and interwoven filaments, fibrals, and plate-like formations of the hydration product. It is this type of bond which is considered to provide the greatest strength in the paste material.[12]

Both of these bonds, however, are highly dependent upon the amount of surface area which is in contact between the various hydration products and the distance of separation between these products. This surface area contact is a function of the amount of void space created by the depletion of the surrounding water in the hydration process. In this manner, it is the void space caused by the depletion of water which has the most significant impact on the strength of the matrix material through the formation of what is termed porosity.[3,12,13,14]

This is the reason for the relationship between a lower water-cement ratio and a higher resulting compressive strength in a normally prepared portland cement material.

Excluding the larger void spaces which occur due to improper and insufficient consolidation in a portland cement material, there are two types of voids or pores which develop in a paste material during the hydration process which contribute to the total porosity of the matrix. One type, gel pores, are the void spaces that exist within the cement gel product as interconnected spaces or voids between the fibrous, interwoven filaments of the gel product. In normally hydrated cements, gel pores are regarded as an integral part of the hydration product and typically occupy approximately one-fourth the total volume of cement gel which is formed.[12,13] As the hydration process proceeds and the cement gel increases in volume, the gel pore space will increase by the same degree but, however, continue to occupy approximately one-fourth the total volume of hydration product formed. Due to the inherent nature of gel pores in the hydration product, they are considered to have less influence on the engineering properties of a cementitious material than other types of voids and pore space.[3,12,13,14]

The other type of pore space has a much greater effect on the engineering properties of the cement paste material. Termed capillary pores, these pores are the larger of the void spaces present in the matrix material and are defined as the space which exists between the various hydrating cement particles. This differs from the gel pores which are defined as an integral part of the cement gel product. The

influence of the initial amount of water used in the formation of the matrix material on the resulting capillary porosity is extreme as increasing amounts of water will serve to further separate the individual cement grains making sufficient particle surface area contact increasingly improbable.[3,12,13,14]

The total amount of capillary porosity present in a matrix material is, therefore, dependent upon the amount of water used in its formation and the consolidation effort applied to the freshly mixed matrix. The total volume of the capillary porosity in a paste material decreases as the volume of the hydration product increases, depleting available water from the surrounding vicinity and replacing that space with cement gel material.[3,12,13,14] Because of this, it is highly beneficial to use extremely low water-cement ratios and to effectively eliminate as much of the potential void space between the individual cement particles before the hydration process begins. In the production of ultra-high strength cementitious materials the elimination of substantial amounts of this type of porosity is a primary concern and the high pressure compaction of anhydrous portland cement is particularly well suited to meet this objective.

The Strength - Porosity Relationship

The discussion concerning the nature of strength in portland cement material in the preceding section was primarily aimed at developing an understanding of the relationship between the hydration process and porosity. The basic concept underlying much of this

discussion was that the strength of a portland cement material is dependent upon the amount of porosity present in the system. At this point it is appropriate to explore this concept in greater detail.

As with any solid material, the physical properties of a portland cement material depends on its internal structure and porosity. Compressive strength is one of these properties. The relationship of compressive strength increasing as porosity decreases and material density increases has been supported numerous times by various researchers.[11,15,16,17,18] As a summary of the state-of-the-art concerning the strength - porosity relationships in portland cement materials, the following discussion will focus on four relationships which have been developed from this previous work.

Perhaps the most profound relationship concerning strength and porosity and the one which established the framework for cement strength - porosity research in this century was developed by Abrams in 1919.[19] Abrams developed the concept that the strength of a portland cement material is inversely related to the initial water-cement ratio which was used in its formation. In other words, as the water-cement ratio used in a mix was lowered, the resultant strength would be greater. This point is what the discussion in the previous section was based upon, that is, an abundance of water in a mix can have detrimental effects on strength by producing excess porosity in the form of capillary pores.

This concept was later expanded by T.C. Powers to the point of development of a minimum water-cement ratio needed for the complete hydration of all cement material with ratios in excess of the minimum

providing water which will only form deleterious capillary porosity and reduce strength.[13] Thus the relationship between a lower water-cement ratio and in essence lower capillary porosity resulting in a higher compressive strength was developed.

Another strength, porosity, water-cement ratio relationship which has significant meaning to concrete strength technology is based on the concept that the strength of a portland cement material is proportional to the increase in the gel/space ratio. Developed by Powers and Brownyard, this relationship states that the strength of a material is directly proportional to the concentration of the hydration products developed in the space available for the hydration to occur.[13]

This relationship is expressed in the form:

$$f'c = (f^{o}c)X^{n} \qquad (1)$$

where f'c is the predicted strength of the paste material,

$f^{o}c$ is the intrinsic strength of the cement gel (i.e.X=1),

X is the ratio of gel volume to gel volume plus capillary porosity (Gel/Space Ratio),

n is a constant depending on the cement characteristics and typically is a value in the range of 2.6 to 3.0.

The purpose of this equation is to relate the compressive strength which can be achieved to the actual hydration product formation and intitial water-cement ratio. Upon the combining of the

mixing water with the anhydrous cement material, the quantity of available space for hydration product formation is the amount of space occupied by the mixing water. Upon a certain degree of hydration however, the available space becomes the volume of hydration product plus the capillary pores that exist. The important point here is the relationship between a lower volume of capillary pores and the corresponding increase in the concentration of the hydration product in the space available for the hydration to occur. This would then mean an increasing amount of surface area contact among the many components in the hydrating matrix. Therefore, it can be deduced that strength in a portland cement material increases as the surface area contact between the various hydration products increases.

As with other solid materials, as the porosity of a portland cement material decreases and the strength increases, the matrix material will behave as a brittle material and its mechanical properties such as strength may be compared with those of ceramic materials.[11] With the high pressure compaction of anhydrous cement, this is exactly the case as the porosity is significantly reduced and ultra-high strengths are obtained. In this respect then it is appropriate to utilize a relationship which defines the porosity - strength relationship for various types of brittle, low porosity, high strength materials.

This relationship is:

$$S=S_o e^{-kp} \qquad (2)$$

where S is the predicted strength,

So is the strength of the material at a condition of zero porosity (intrinsic strength),

P is the fractional porosity at a given time, and

K is a constant which depends on the system being studied.[11,13]

This relationship is well suited to portland cement materials because of the wide range of porosity values for which the expression holds true and also because as the porosity level approaches very low values, the compressive strength increases tremendously.

While these relationships are excellent models of the general relationship between porosity, hydration, and strength for normally prepared cement paste materials, there exist other factors such as cement composition, morphology and bonding properties of the hydration products, which also affect the strength of a set* cement paste.[18] This is particularly true in the case of highly compressed, low porosity portland cement materials.

In the extremely low porosity conditions for materials produced by high pressure compaction there exist other factors which effect the resulting strength. As noted, an excess in porosity is the major contributor to a reduction in strength and as such will generally surface as the controlling strength- determining factor in materials with higher porosity levels. According to work by Relis and Soroka

* Set refers in this case to a hardened paste which has experienced considerable hydration product formation and possesses a substantial amount of strength.

there are other factors which have been found to produce excellent strength correlations for the extremely low porosity range produced by the high pressure compaction of anhydrous cement powders.[18]

This work is based on the rationale that the three components of a set cement, namely porosity, hydration product and unhydrated cement are interrelated and a change in one will result in a change of the other two. If the assumption is made that both porosity and the amount of hydration product present in the matrix are influential to strength, then in a low porosity system the strength of a set cement will increase with an increase in the volume of hydration product and a decrease in the porosity level. According to their work it is possible for the unhydrated cement grains to be stronger than the C-S-H gel hydration product formed which would mean that a large concentration of unhydrated cement material would also contribute to compressive strength.[18]

From this concept, the following relationship was found to have an excellent correlation between its value and the compressive strength produced.

$$(W^*_{hp} \times G^*_c)/P_f \qquad (3)$$

where W^*_{hp} is the weight concentration of hydration product formed,

G^*_c is the weight concentration of unhydrated cement,

P_f is the final porosity in fractional form.

Relating this relationship to compressive strength for the compressed cement material, excellent results have been obtained for up to 28 days of curing.[18] For periods greater than 28 days it appears that other age factors come into effect and this relationship does not correlate well with actual strengths.

In summary, a number of conclusions can be drawn concerning the relationship between compressive strength and porosity conditions in a portland cement product. They are:

1. Porosity is inversely related to strength. When porosity of a portland cement material is decreased, the compressive strength increases. (water - cement ratio law)

2. The water content in a mix, if in excess to that needed for hydration, can have deleterious effects on strength.

3. The formation of hydration products produces higher strengths by decreasing porosity and increasing the amount of surface area contact between the many particles composing the matrix.

4. In most instances, porosity has the majority influence in strength development for portland cement materials, however at extremely low porosity levels such factors as the concentration of unhydrated cement material may have considerable influence on the strength which is achieved.

From these conclusions, the importance of the high pressure compaction in the production of a high strength material is understandable. According to the preceding discussion, to obtain a high compressive strength material, the objective is to modify the microstructure of the matrix material in such a way that the capillary

porosity is reduced to a large extent. If the compaction is performed on an anhydrous cement material, the effects of excessive amounts of water are eliminated as the necessary moisture for hydration is imbibed through porosity that exists between the cement particles. Thus high pressure compaction of anhydrous cement material is an attractive method of producing a higher strength material.

The Process of High Pressure Compaction

The objective of high pressure compaction is to increase the surface area contact and consequently reduce the void space between the powder material so that interparticle forces may develop which will produce high strengths.[20] The mechanisms which commonly accomplish this objective are particle sliding or rearrangement, plastic and elastic deformation of the material being compacted and particle fragmentation.[21] Fig. 1 shows a schematic representation of these three processes.

In the process of applying high pressure to a particulate material, there are two interrelated stages which occur. These are 1) the process by which the particulate material will attain a state of closed packing or low porosity level and 2) the process by which the particulate materials cohere between themselves resulting in a high strength material. Commonly this second process is accomplished by various methods of adhesion and aids significantly in producing the stable high strength condition in the low porosity material.[22]

The first stage in the compaction of particulate material is the

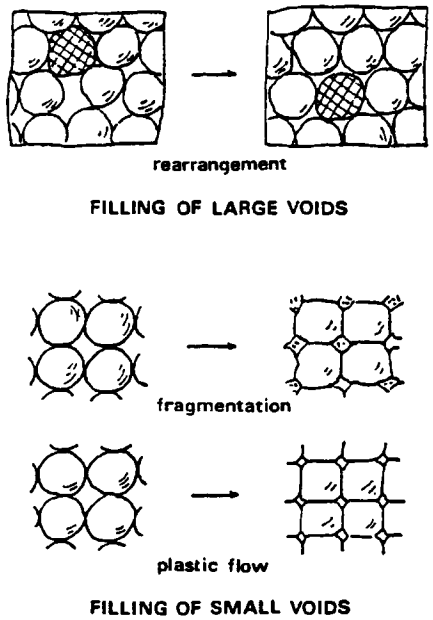

Fig. 1 [22] Schematic diagram of compaction process in particulate material

general rearrangement of the particles to obtain a condition of closer packing or lower porosity. This occurs as the material chooses a "least path of resistance" and the material particles slide next to and in between each other. The elimination of the larger voids is the primary occurence at this stage. At this point, much of the compaction energy input is dissipated in overcoming the interparticle frictional forces which exist at material contact points. For fine ceramic powders, it has been found that under the comparatively low pressures induced in this initial stage, one-half to one-third of the total sample volume reduction will occur.[20,22]

The second stage in the compaction process occurs as increasing pressures are applied and depends on the elasticity and plasticity of the compressed powder particles in further obtaining a reduction in voids. Commonly plastic deformation and fracture of the material particles occurs during this stage. Under the higher applied pressure associated with this stage, the particles will either fracture or deform plasticly and flow into the available void spaces to reduce the void spaces remaining from the first stage. This action results in increased surface area of interparticle contact and therefore increases the area available for interparticle bonding. Should fracture occur in the particles, large reductions in sample volumes may occur, a common characteristic of brittle materials. Depending on the particle material characteristics, substantial amounts of particle interlocking due to geometrical properties may occur.[20,22] For this reason, rough textured, sharp, angular particles are preferred over smooth-surfaced, spherical particles when preparing compacts with high pressure.

In reality these various stages are occuring simultaneously throughout all portions of the powder material as the compaction pressure is applied. This simultaneous process will occur until the porosity and void content of the sample approaches non-existence and the compact density approaches the actual density of the solid material. Throughout both stages of the compaction process, elastic compression of the trapped air as well as the particulate materials will occur. As the pressure reaches extremely high amounts, the void - porosity system will become fully disconnected, consisting only of

very minute isolated pores throughout the system. As this destruction of the void - porosity system occurs, extremely high internal gas pressures also develop. For this reason, as well as the storage of elastic energy due to the elasticity of the compressed material, the release of a good deal of stored energy will result once the compaction pressure is released. Thus, release of the compaction pressure on the compact sample should be done gradually in order to eliminate the risk of total compact disintegration.[20,22]

Also during the process of compacting particulate materials, several other factors come into effect which influence the obtaining of optimum results. Chief among these are the characteristics of the material such as particle shape, texture, and strength as well as plasticity and elasticity characterisitcs. Studies have shown that as the reduction in the larger voids occurs, an increase in the number of smaller voids also occurs. Initial gradation of the material also has a significant effect on the amount and type of void reduction which occurs. Studies have shown that compaction of single size particles results in higher amounts of particle fracture. However with the inclusion of a range of sized particles, researchers have found that the amount of fracturing diminishes significantly.[21]

The actual type of machinery used also affects the ultimate goal of producing the highest density possible due to the devolopment of frictional forces. Frictional forces commonly develop along both the walls and the top and bottom of the pressing die. These frictional forces produce shear forces at the wall particle interface producing

non-uniform pressure distributions. Theoretically, the compacting pressure should be transmitted throughout the material providing uniform pressure and constant density throughout a sample. This is not the case however as the frictional forces developed lead to various points and planes of weakness in a pressure produced compact. The use of various lubricants in eliminating this frictional effect are commonly used; however, these may have a detrimental effect on the strength produced.[20,22] Fig. 2 shows a typical pressure contour pattern which may develop in a compact being prepared by high pressure compaction.

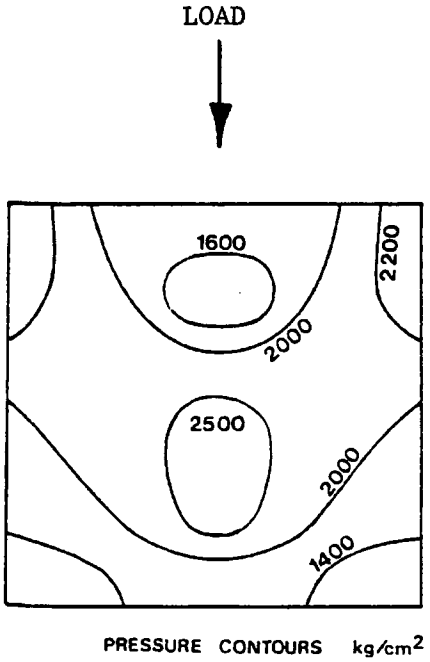

Fig. 2[22] - Typical pressure contours which develop in prepared compacts

High Pressure Compaction of Portland Cement Powders and Pastes

As mentioned previously a thorough search of the literature yielded very little information on the high pressure compaction of portland cement concrete material. The search, however, provided substantial amounts of information concerning the high pressure compaction of portland cement pastes and anhydrous powder.[15,16,17,18,23,24,25]

From the literature surveyed, the high pressure compaction of both cement pastes and anhydrous cement is typically performed with the aid of a specifically designed die or mold.[16,24,26] High pressure compaction of a material with a die is a simple operation which consists of the placement of the anhydrous powder or cement paste in the die chamber and the insertion of a plunger into this chamber for actual compaction of the material upon application of a compacting load.

The die or mold chamber where the compaction process occurs is generally formed in one of two ways. One method is the use of one solid piece of extremely hard, high strength steel which has a chamber tooled into it. A second method is the use of various tooled segments which are fabricated in such a way to form a chamber and held to together by an external ring or high tensile steel bolts. A compacting ram is then inserted into the chamber for the application of the compaction effort to the powder material. The degree of powder compaction can either be limited by the stroke of the ram or by the level of compaction effort applied.[16,24] Dies have been used in

which the compaction pressure was applied from both a single vertical force or from dual forces acting from opposite sides of the sample being prepared.[16,24,26] With the use of a segmental die the prepared compact is removed from the die generally by disassembly of the die itself. This differs from the other method where the prepared compact must be extruded from the die.[16,17,24]

Due to the extremely high production pressures to which the die equipment is exposed, it is necessary to use high tensile strength materials in their construction. To eliminate as much of the friction as possible between the plunger and the die walls it is necessary to tool these surfaces extremely smooth in addition to the utilization of hardened steel.[16,24] The tolerance of fit between the plunger and the die chamber is also critical as, with addition of the compaction pressure, powder in the chamber will "escape" around the plunger. This will also lead to an increase in the friction present between the plunger and the die walls. In many instances this friction developed is reduced with the use of special lubricants such as stearic acid or mold oils.[15,16,17]

Generally when high pressure compaction is involved, whether it be an anhydrous cement, paste or mortar material, the physical size of the specimen produced is small.[15,16,17,23,24,26] The reason for this is twofold. One, the use of smaller specimens permits the use of smaller capacity production equipment such as hydraulic presses and two, the use of smaller specimens facilitates the inexpensive production of a greater number of specimens in a shorter period of time and under stricter quality control conditions.[24] Compacts

beginning on the order of one cubic centimeter in size on up are commonly produced.[24] The geomtrical shapes produced include cubes, cylinders and disks.[16,23,24,26]

From the literature surveyed, the level of compaction pressure was quite varied and seemed to depend on the general intent of the individual laboratory programs and the limits of the available equipment. The lowest compaction pressure in the literature was 1500 psi (10.3 MPa) while the highest appeared to be 116,000 psi (800 MPa).[16,23] The rate of loading was seldom mentioned in the literature. Two values which were reported however were 4,380 psi (30.2 MPa) per minute in one instance and 11,220 psi (77.4 MPa) per minute in another.[16,24] Needless to say, increases in compaction pressures resulted in decreases in the initial porosity condition of the prepared compacts in all cases.

As mentioned in the previous section, during the actual compaction of the material, various forces develop within the material which can be harmful to the proper formation of the compact. For this reason it is thought to be advantageous to hold the production pressure for a period of time once the desired pressure level has been reached.[15,16,24,25,26] From research conducted in this area, it appears that holding the production pressure for an excessive amount of time has little effect on the initial porosity of the compact or its ultimate compressive strength.[15,16] From these conclusions, typical holding times of one to three minutes are commonly used with excellent results obtained.[24,26]

As mentioned earlier, a search of the literature provided substantial information on the high pressure compaction of both anhydrous cement and cement paste materials. Excellent results have been achieved in regards to producing high compressive strength materials. There do appear to be different advantages between the two however.

For an anhydrous material, the quality control of the prepared compacts is much better than for a paste or mortar material.[24] This stems from the fact that if an anhydrous material is used, no complications are involved with the addition of the correct amount of water and the necessary mixing of the two. Calculation of the initial porosity or the porosity of the prepared compact before hydration has progressed is also simple as the specific gravity of the powder, bulk weight and and bulk volume provide this value. The mixing water content in this type of compact is essentially the volume of the void space within the cube which would be filled with water upon immersion. In this respect then, the initial porosity of an anhydrous compact is also a crude measure of the water-cement ratio for a prepared compact.

For a paste material, a unique phenomenon makes it an attractive material for high pressure compaction tests. In the previous section, the importance of lowering the water content in the matrix material was discussed and it seems to be a safe conclusion that the addition of no water would produce a stronger material upon full exposure to moist curing conditions. Such is not the case. Researchers have found that in the high pressure compaction of cement

pastes, there exists an optimum water content for each production pressure. This is defined as the water content at which water from the paste material was not pressed out as a specific compaction pressure was applied.[23] This optimum level decreases as the compaction pressure increases.

The optimum water content is unique in that compacts prepared with increasing amounts of moisture from zero to the optimum level, exhibited an increasing density condition, i.e. less porosity, while with compacts prepared with water contents above the optimum level, a decrease in the achieved densities at the same compaction pressures were experienced. The resulting strengths exhibited this same phenomenon at early ages due to the increased density and lower porosity. However, over time the effects of this initial condition become less evident due to the formation of hydration products.[15,23] In summary, it appears that with the high pressure compaction of portland cement materials, there is a certain optimum water content which optimumly acts as a lubricant between the individual particles of cement resulting in denser, lower porosity materials at a given production pressure.

The characteristics of the cement also have an effect on the results in the high pressure compaction process. It has been shown that cements with lower specific surface areas (coarser material) compacted at the lower production pressures, produce compacts of lower apparent specific volumes.[16] This means that for the compaction of coarser materials, the resultant porosity is lower than that of the

finer materials. This relationship is more evident for the lower production pressures than at the higher production pressures. Concerning the importance of particle size distribution, it appears that a mix combination of two-thirds coarser particles with one-third finer particles will result in the lowest porosity condition for a given production pressure.[16] A similar coarse aggregate to fine aggregate ratio is needed for a well graded concrete mix.

In the formation of portland cement compacts there are several problems which are typically encountered. One such problem is a difficulty in controlling the porosity and dimensions of the prepared compact due to expansion upon the removal of the compaction pressure and subsequent removal from the die. This is typically overcome by taking the expansion into account and adjusting the quantity of initial material inserted into the die. In this way fairly constant results can be obtained. This expansion is primarily dependent upon the characteristics of the powder material.[16,24]

A second problem is what is termed the "detachment of end caps". This refers to the formation of small cracks around the perimeter of the prepared compact at the surface where the plunger applied the compactive pressure. Generally these cracks extend into the compact and tend to intersect at a certain distance internally in the compact. This results in a "cap" like failure on that surface of the compact.[16]

Several causes of this failure are possible. These are:

1.) Non-uniform powder distribution in the die chamber before the compaction pressure is applied resulting in small points of higher degrees of compaction which could possibly expand more in relation to

the surrounding powder resulting in non-uniform expansion upon release of the compaction pressure.

2.) Insufficient rigidity in the material of the die wall may allow the compacted material to expand radially during compaction causing a shear failure at the point of pressure application.

3.) If the radial pressure that develops between the compact and the die wall does not dissipate as the compaction pressure is released, then the compact will be left in a state of biaxial compression with the die walls and the third axis or axis of application of the compaction pressure will be in a state of tension.

4.) If upon the removal of the compaction pressure a non-uniform expansion occurs due to friction between the die wall and the powder, the outer surface of the compact might develop a crack.

5.) Due to compressed pockets of air, the compact may burst upon removal of the compaction pressure through the release of compressed air.

It appears that this "cap" failure is minimized by the use of lubricants on the die walls as well as the ram - powder interface.[16]

Before the hydration process begins, the compact is stable due only to the adhesive forces present between the particles. These forces are primarily dependent upon the degree of compaction attained within the compacted material, the particle size distribution of the material and chemical composition of the powder.[16]

Curing conditions for compacts prepared in previous work vary as much as the production pressures that produced them. Since much of

the work done to date concerning the high pressure compaction of portland cement materials pertains to the development of early strength predictions, curing conditions which involved high temperature curing solutions were used.[15,16,17,24,27] This discussion will not pertain to these systems.

Generally, the most common curing regime for simple compressive strength determinations is the immersion of the compacts in de-ionized or distilled water for the required period of time.[15,17,23,24] The temperature of this water was in the range of 68 to 70 F (20 to 25 C) for the literature surveyed. As discussed in The Nature of Strength In A Cementitious Material section of this report, one product of the hydration process is the production of calcium hydroxide. In high pressure prepared compacts, due to the extreme internal pressures which develop, the calcium hydroxide leaches from the compacts. Some researchers believe that if the compacts are cured in a saturated lime solution, zero to minimal leaching of the calcium hydroxide produced will occur.[28] Further studies have proved otherwise.[28]

Testing conditions are also numerous and varied. From the literature, the loading rates varied from 4,350 psi (30 MPa) per minute to 8,000 psi (55 MPa) per minute.[15,16] In one instance, grinding of the surface was done to obtain a smooth surface for testing.[16] In some of the work, special devices were employed for the failure process due to the small size of the compacted specimens to be tested.[24] There is no record of end-capping or other special preparation for the compacts tested other than the grinding suggested above.

The Hydration of Prepared Compacts

From the discussion on the nature of strength in a cementitious material, the point was made that both porosity and the formation of hydration products are key ingredients to the development of strength. As in normally prepared cementitious material, the hydration process in pressure prepared compacts proceeds as long as free water is available. The process of hydration is more complex than with that of a normally prepared material due to the extreme reduction in porosity which occurs.

Generally, the relationship is such that as the compaction pressure decreases, the resulting compacted material will have a higher degree of hydration.[23] In other words, as the initial porosity of the material increases, the degree of hydration also increases meaning that a greater volume of strength producing hydration product is formed and the resulting decrease in porosity is greater.[22,29] The greater the initial porosity level, the greater the degree of structural change within the compact from curing.[29] Compacts prepared at higher compaction pressures, with the resulting increase in structural density, exhibit slower rates of hydration and hydrate at later ages.[29]

However, for the condition of lower initial porosity where less hydration product is later formed, on an effective influence per unit of material basis, the hydration product that is formed has a far greater effect on producing strength. This is primarily due to the

relatively large increase in surface area contact between the various products in the system. The resulting percentage change in total porosity is also greater for the lower initial porosity conditions.[23] This process whereby a unit formation of hydration product in a lower porosity material has a much greater effect on strength than at a higher porosity condition is referred to as an increase in the specific binding capacity of the hydration product formed.[29]

Concerning the formation of hydration products, it has generally been determined that no new forms of hydration product are produced as a result of the high pressure compaction process.[26,29] Upon visible inspection with the aid of sophisticated equipment, products of C-S-H, calcium hydroxide and other common hydration products are clearly visible, however in decreasing amounts as the initial porosity decreases.[29] The characteristic fibrous growths and rosettes of the C-S-H materials as well as the characteristic plate-like appearance of calcium hydroxide become less evident as the production pressure increases.[26] In general, the greater the production pressure, the more amorphous in appearance is the structure of the compact. The rate of hydration product formation is also influential on the microstructure of the compacted material.[26]

Due to the extremely high concentration of cement material present in pressure prepared compacts, it is plausible to suggest that this high density condition may have negative effects on the hydration process. Chief among these are extremely high temperature evolutions leading to thermal cracking, deleterious expansion of the compact if hydration is allowed to proceed at an uncontrolled pace and the

formation of a limiting mechanism which would hinder the hydration of material deeper within the compact. Concerning the first two of these potential problems, subsequent laboratory tests have shown that neither extremely high temperature evolutions or uncontrolled expansion occurs for smaller sized compacts, but as the size of the compact increases, these effects become more influential.[16] There is however some merit to the concept of a limiting mechanism.

The theory supporting this concept is that due to the low porosity condition in the prepared compacts, permeability of water necessary for hydration to the inner portions of the compacted material is limited. This density is further inhibited by the formation of hydration product toward the outer portions of the compacted material and thereby virtually eliminates the exposure of the unhydrated material within the compact to necessary water needed for hydration.[16,28,30]

In this regard, the hydration reaction has been described as occurring in one of two stages.[28] The first stage is described as hydration occurring through solution with the hydration product precipitating on the surface of the cement grains. This type of hydration occurs upon the initial exposure of the prepared compact to a curing solution. The second stage is referred to as hydration through diffusion as the hydration is occuring at the cement - gel interface and the necessary moisture must pass through the cement gel which has developed around the individual cement grains.[28] It has been shown that rate of take-up of water decreases with decreasing

initial porosity and also with increases in curing duration.[16]

A considerable amount of work has been performed in the area of hydration product formation and properties in pressure compacted pastes and anhydrous material.[18,28,30] It has been shown that the average density of the hydration product in high pressure prepared material increases with the increase in the degree of hydration and the decrease in the initial porosity.[28,30] This relationship is a result of pressure build-up due to volume constraints which develop in the compact as the formation of hydration product occurs. It has been shown that the hydration product produced by diffusion are denser than that formed by hydration by solution.[28,30] The relative amount hydration product formed by diffusion increases with an increase in degree of hydration and a decrease in porosity.[28,30]

Strength - Porosity Considerations of Prepared Compacts

As developed in an earlier section, there is an inverse relation between strength and porosity. For prepared compacts of portland cement material, this relationship has been shown in practically all work done to date.[15,16,17,25,26,30] When combining this relationship with the formation of hydration product relationship just discussed and two important general rules become apparent. These are 1) compressive strength increases with a decrease in porosity, and 2) conditions of greater initial porosity will allow for the greater formation of hydration product and because of this, with increasing curing duration compacts with higher initial porosities will show a greater amount of strength gain than compacts with the lower initial

porosities.[15,16,25] This relationship between porosity and strength for an initial porosity range from 20 to 35 percent appears to exist as a simple linear relation.[15,16,25] Elimination of as much initial porosity as possible by high pressure compaction is the most influential method of producing a high strength material at all subsequent ages of curing.[15,16,25]

The level of initial porosity obtained in compacts of portland cement material is dependent upon the amount of compaction pressure used and the particle size distribution of the material.[16] In regards to the initial porosity level, 20 to 35 percent is a general range of porosities which is often achieved for laboratory study of the effects of high pressure compaction on compressive strength.[15,16,25,30]

Final porosity or the porosity present in the compacted material upon completion of the desired curing duration is a function of the initial porosity and the amount of hydration product which has developed.[30] It has been shown that, assuming the specific volume of evaporable water is unity, the final porosity of a compacted material can be predicted somewhat by the quantity of evaporable water at 220 F (105 C).[28] This prediction accounts for a large portion of the gel porosity as well as the capillary porosity which is present in a hydrated material.

Some observers feel, however, that at the the temperature used to determined the "total" free water content, some free water in addition to chemically combined water still exists within the paste which

contributes to the total porosity content.[12,13] For this reason, another procedure is often used whereby the additional moisture not eliminated at 220 F (105 C) yet contributing to the total porosity content is determined. This procedure is a loss on ignition test at 1800 F (1000 C).[15,16] With this procedure, in addition to the free water not eliminated at 220 F (105 C) various forms of water existing between the free and chemically combined states is also eliminated.[12]

Final porosity levels which have generally been attained in the laboratory programs range from 10 to 30 percent.[15,16,23,27,28]. As mentioned earlier, lower final porosity values have been achieved with addition of heat to the process of compaction and subsequent curing.[27,31]

The level of compressive strengths achieved in the laboratory studies to date is dependent on many factors, however excellent results have been achieved for various combinations of compaction pressure, curing duration, porosity level and material selection. Because of the numerous variables which effect the strength of the compacts, it is very difficult to correlate and enumerate the strengths achieved from the various studies. It is sufficient to say that compressive strengths as great as 40,000 to 50,000 psi (275 to 345 MPa) can be obtained in 28 days in a simple laboratory situation of high pressure compaction and moist curing of small compacts.[16,17] The point also needs to be made that in many cases the extremely small size of the test specimens has tremendous influence on the actual compressive strength values achieved.

Although not relevant to the study at hand, it is important to

point out that compressive strengths approaching 95,000 psi (655 MPa) in 28 days has also been reported using a procedure whereby the material is simultaneously heated while the compaction pressure is applied.[17,18,26] This procedure, termed hot-pressing, causes a more rapid hydration process which in turn causes a substantial material rearrangement within the microstructure of the material.[17] A porosity of 1.8 percent has been achieved by this method and is considered to be the closest approach to zero porosity or theoretical density for portland cement material attained to date.[17] It has been shown that, in terms of producing increased strength at the time of preparation, increasing the temperature with this technique will have a more positive effect on strength than increasing the compaction pressure.[17]

From previous work in producing high compressive strength materials through high pressure compaction and the associated influence of porosity, a number of prediction equations have been proposed to predict the strength of prepared compact samples from their porosity level.[15,16] From this work, theoretical ultimate strength predictions of approximately 72,000 psi (496 MPa) at a zero porosity condition have been predicted by the compaction of anhydrous powder.[15,16] Because of the initial anhydrous condition, this value is believed to be a prediction of the strength of anhydrous clinker material.[16] The bond created in a portland cement material is a hydrate bond however and not a ceramic bond as exists in clinker material. Because of this, it appears that the ultimate strength of

a hydrated material may be much greater, depending primarily on the amount of initial porosity reduction which can be obtained through high pressure compaction.

The Nature of Pozzolanic Reactions

As mentioned at the outset of this discussion, high pressure compaction is a mechanical means of reducing the porosity and ultimately increasing the strength in a portland cement product. A pozzolanic reaction is a chemical means of reducing the porosity and increasing the strength in a portland cement product.

A pozzolanic reaction is a chemical reaction which occurs whereby available siliceous materials react in the presence of water with the free lime or calcium hydroxide ($Ca(OH)_2$) produced by the hydration process to produce calcium-silicate-hydrates (C-S-H). In equation form this reaction may be written as :

$$CH + S + H \Rightarrow C\text{-}S\text{-}H$$

where: CH represents calcium hydroxide,

S represents silica dioxide,

H represents water, and

C-S-H represents calcium-silicate-hydrate formed.[13]

The effect of a pozzolanic reaction is to react with the calcium hydroxide formed during the hydration process to form stable, strength producing C-S-H at the expense of the weaker $Ca(OH)_2$ in the matrix material.

The free lime, in the form of calcium hydroxide , is produced by the hydration process of the cement and is essentially a waste product which is produced when the calcium silicates (C_3S and C_2S) hydrate to form calcium-silicate- hydrates and calcium hydroxide. The formation of this product as a hydration product in this manner produces a crystalline material which is very precise in its stoichiometry whereas that of the calcium-silicate-hydrates is not well defined. The morphology of these calcium hydroxide crystals is dependent on outside influences such as hydration temperature and various admixtures and can exist as large flat platy crystals, large thin elongated crystals and all variations in between.[12,13]

The important thing about calcium hydroxide is the fact that its crystals only grow in availble free space and in this respect act to eliminate detrimental capillary porosity to a certain extent. Calcium hydroxide crystals may occupy as much as 20 to 25 percent of the paste volume.[13] The lime itself is not cementitious in nature, and the addition of a pozzolan to a portland cement material results in strength increases by transforming the non-cementitious lime into cementitious calcium-silicate-hydrates. In this way a pozzolanic material increases the concentration of strength producing calcium-silicate-hydrates at the expense of free calcium hydroxide.

Fly Ash as a Pozzolan

To create a pozzolanic reaction in the form listed above, one must provide the available free lime with a pozzolan in the presence

of water. A pozzolan is defined in ASTM C618 as "a siliceous or siliceous and aluminous material which in themselves possess little or no cementitious value, but will in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties".[32] Fly ash or the residue remaining from the combustion of coal is just such a material. Fly ash, a material which is available in great abundance as it is currently considered a waste product by the power industry which generates millions of tons per year, is primarily composed of silica, alumina, ferric and calcium oxide compounds.[33] Depending on the type of coal material from which it is produced, fly ash typically contains various amounts of these oxides.

ASTM C618 defines a fly ash as a pozzolan under one of two classes.[32] A Class "C" fly ash is one which possesses pozzolanic and cemetitious properties normally produced from lignite or subbituminous coal and generally contains a considerable amount (>10 percent) of lime which provides for the formation of additional cementitious properties when exposed to water. A Class "F" fly ash is classified as one with pozzolanic properties normally produced from anthracite coal or bituminous coal and generally contains neglible amounts of lime. The Class "F" fly ashes are considered low lime fly ashes while the Class "C" fly ashes, which have been known to contain as much as 40 percent lime in addition to the silica, are considered high lime fly ashes.[34] In this regard, Class "C" fly ash is then the more attractive pozzolanic material as it is chemically

bound to available lime, silica, alumina, and iron and exhibits cementitious properties similar to that of portland cement.[34] Both classes of fly ash have seen considerable use in the concrete industry.

Concerning chemical requirements, ASTM C618 has established acceptable levels for the oxide concentration in the various fly ashes for use in a concrete mix.[32] A Class "F" fly ash must have a minimum of 70 percent by weight the combined oxides of silica, alumina, and iron. A Class "C" fly ash must have a minimum of 50 percent by weight the combined oxides of silica, alumina, and iron. Since fly ash is a residue of combusted coal, there exists a certain amount of unburned coal or carbon particles. ASTM C618 has established a 6 percent maximum by weight for both classes of fly ash.[32]

The fly ash, having been exposed to extremely high temperatures at the time of its formation and because of its silica content, has many of the same qualities of a glass-like substance.[33,34] It is during the exposure to the extremely high temperatures in the combustion furnace that tiny droplets or spheres varying in size from 1 micron to 1 millimeter are formed which are a combination of the various oxides which compose a fly ash particle. Fig. 3 shows an excellent example of Class "C" fly ash particles as photographed by a scanning electron microscope and magnified 3000 times. Upon cooling these spheres become essentially a glass material which is very stable both chemically and physically. It is these particles which are composed of the silica, alumina, iron and calcium oxides, the main

ingredients necessary for a pozzolanic reaction.

Particle size fineness and pozzolanic activity index are two of the physical characterisitics of a fly ash which are used as criteria by ASTM C618 when considering a fly ash for use as a pozzolan.[32] An acceptable fineness of the fly ash is obtained when a maximum of 34 percent of a fly ash is retained when wet sieved on a No. 325 (45 micron) sieve. The pozzolanic activity index is an evaluation of the contribution to be expected from the given fly ash to the long term strength development of the concrete. It is a measure of the reactivity of the fly ash with a given cement. The test is performed by preparing a standard test specimen and a specimen with fly ash replacing a portion of the cement. Upon curing for the specified time, both specimens are then tested for compressive strength and the strength of the fly ash specimen is compared to the strength of the control specimen. ASTM C618 defines a fly ash producing a strength specimen greater than 75 percent that of the control specimen as acceptable.[32]

In general, the finer the fly ash, the greater the amount of pozzolanic activity.[34] This is due to the fact that increasing amounts of finer particles means that there also is a greater amount of surface area than that of coarser particles. Since most fly ash is of the coarser variety, the consequential low surface area results in a slower lime-pozzolan reaction in the presence of water. This is the very reason why portland cement products containing fly ash as a pozzolan often exhibit lower early compressive strengths but at later

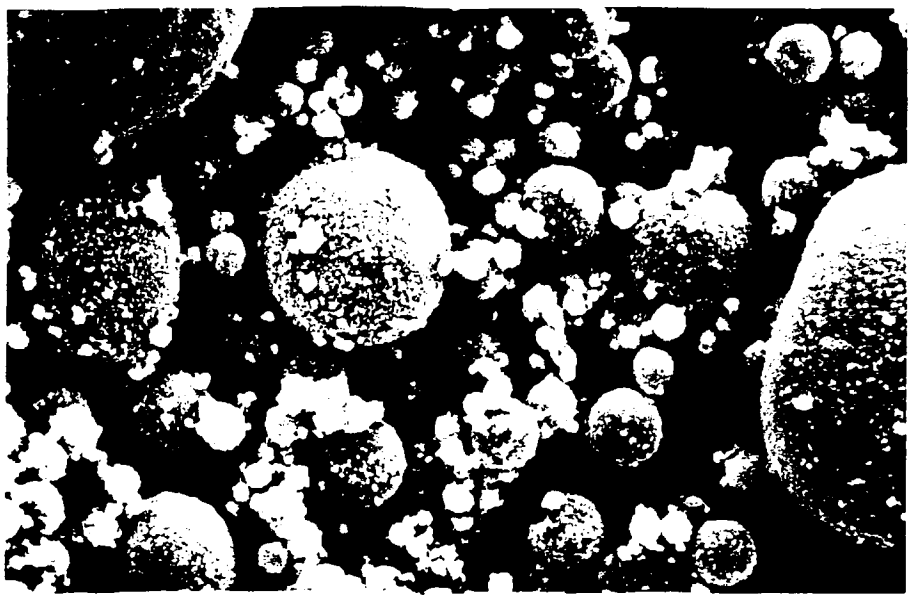

Fig. 3 - Typical Class "C" fly ash material magnified 3000 times

ages produce substantial strength increases. It is this development of higher ultimate strengths which makes the utilization fly ash very attractive as a partial replacement for portland cement.[1,2,5,8,35]

Fly ash is also a very attractive replacement for portland cement in a number of other respects. Since the individual fly ash particles are essentially glass spheres, the workability of harsh or dry mixes of concrete is improved by the addition of fly ash which serves as a lubricant. Because of this better workability, placeability, finishability and pumpability all occur in a mix containing fly ash. The water-cement ratio can also be lowered since much of the water used in a mix is for workability considerations only. Fly ash will also lower the rate of heat liberation due to a

decreased rate of hydration. Impermeability and durability of hardened concrete are also improved.

The main attractive advantage of fly ash materials are as pozzolans. Currently observers consider the use of pozzolans such as fly ash a must in the obtaining of higher concrete strengths.[8,35] Typically rates of replacement of the fly ash for the portland cement range from 15 to 30 percent and can be done on a pound for pound basis provided a high lime, good quality fly ash is used.[1,2,8,34] The optimum amount for replacment is highly dependent upon mix characteristics as well as the reactivity of the fly ash used and for this reason it is difficult state an exact amount of fly ash as optimum from the preceding work done to date.

Summary

The search of the literature failed in providing any information on the combining of fly ash with portland cement and by exposure to high pressure compaction and subsequent curing produce an ultra-high strength material. According to the background discussion presented here, from the favorable results that each of these processes has provided in the past in producing ultra-high strengths individually, it is plausible to suggest that the combination of high pressure compaction reduction of porosity coupled with increased formation of cemetitious products of a pozzolanic reaction into the system might have trememdous success in producing an ultra-high strength product.

CHAPTER III

DEVELOPMENT AND PERFORMANCE OF THE LABORATORY INVESTIGATION

Parameters Investigated

The effects of the following parameters on the compressive strength of the prepared compacts were investigated. The parameters were:

1) calculated initial porosity,

2) percentage replacement of portland cement with fly ash by weight, and

3) curing duration.

The remainder of this Chapter describes the performance of the laboratory investigation into the effects of these parameters on the compressive strength of the miniature compacts.

Development of the Laboratory Program

Materials and Equipment

This study involves the high pressure compaction of anhydrous cement powder with a fly ash material to produce a high compressive strength material. Since the ultimate objective was to obtain a high compressive strength material, the type of materials selected for use were an important consideration. Only one type of cement and fly ash were used throughout the duration of the project.

The cement selected was a Midlothian TXI Type III cement produced by Texas Industries Inc. at their Midlothian , Texas plant. A Type

III cement was selected because of the higher early strength which could be achieved. The fly ash selected was an ASTM C618 Class "C" marketed by the Gifford-Hill Company out of Dallas, Texas. A Class "C" material was chosen because of the high lime content it contained and the resulting high degree of pozzolanic activity which would result when combined with the Type III cement. Complete chemical and physical analysis test results for both of the materials use are included in Appendix A.

The die equipment for this study was designed by Dr. Matti Relis and was developed by him after many years of experience in the area of high pressure compaction of cement powders.[18,24,28,30] Fig. 5 and 6 show an assembled and disassembled view of the die respectively. This particular die is a segmental die in that the four interior segments are tooled for exact fit and held together by a clamp ring bolted to the baseplate. The compacting ram is then inserted into the chamber formed by the tooled segments. The die is fabricated entirely from A-2 tool steel and heat treated to a Rockwell C60 hardness.

For a complete understanding of the actual operation of the die, Appendix B should be consulted. The general principle on which the die functions is that by controlling the depth at which the ram may penetrate into the die chamber, all compacts prepared will be nominally of the same size. This is accomplished through the use of the spacer piece which surrounds the ram. As the compactive force is applied the ram head will come into contact with the spacer disk and

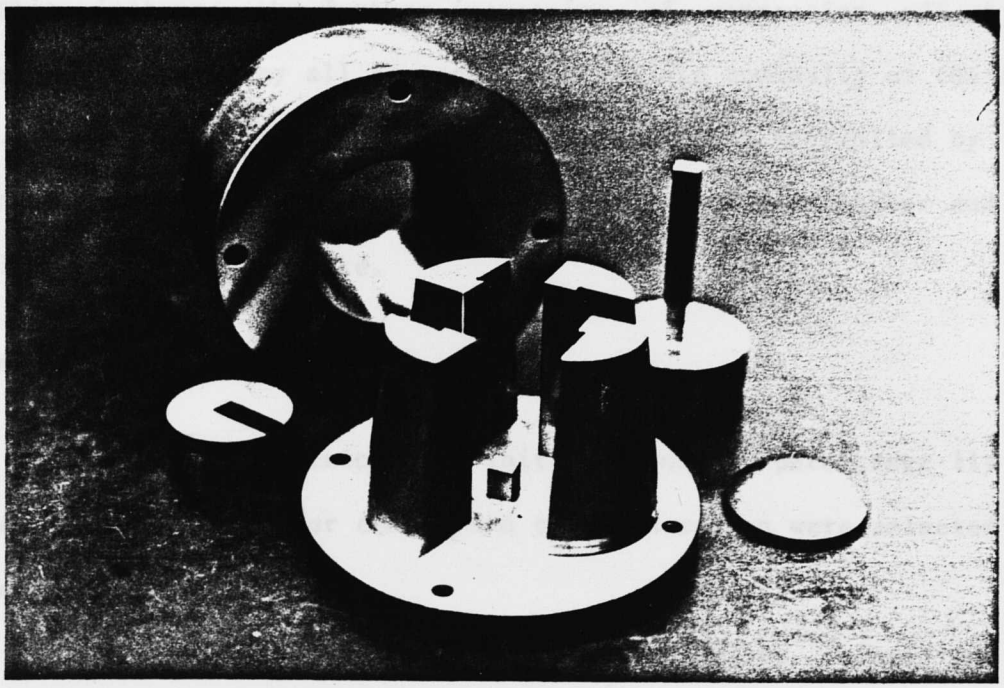

Fig. 4 - Disassembled view of the die used for compaction

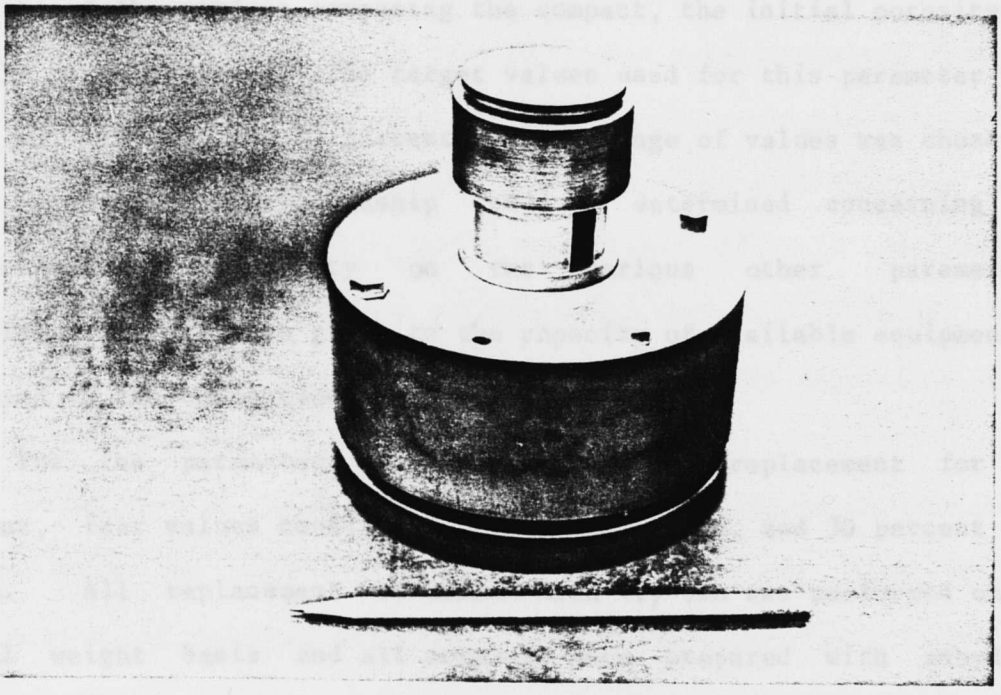

Fig. 5 - Assembled view of the die used for compaction

at this instance the further increasing of compaction effort is halted. In this way all compacts produced are nominally of the same size. Porosity of the prepared compacts is then controlled by the amount of powder which is initially placed in the die chamber and the applied compaction pressure.

Program Design

The parameters of calculated initial porosity, percentage fly ash replacement by weight for cement, and curing duration were selected for investigation.

The parameter of calculated initial porosity refers to the percentage volume of a prepared compact which is void space. Since the compacts were prepared from anhydrous material, using the specific gravity of the powders composing the compact, the initial porosity can easily be determined. The target values used for this parameter were 20, 23, 26, 29, and 32 percent. This range of values was chosen so that a distinct relationship could be determined concerning the importance of porosity on the various other parameters. Consideration was also given to the capacity of available equipment to be used in the compaction process.

For the parameter of percentage fly ash replacement for the cement, four values consisting of zero, 10, 20, and 30 percent were used. All replacement of cement with fly ash was performed on an equal weight basis and all compacts were prepared with anhydrous material.

For the parameter of curing duration, 3, 7, 28, and 90 day

durations were utilized. Although the pozzolanic reaction expected to occur would be slow developing and perhaps require longer durations, these durations were decided upon because of the time frame of the present study and to provide background information for future studies of the pozzolanic reaction in a high pressure prepared compact.

In addition to the possible combinations of the three main parameters, it was also necessary to prepare compacts to be tested in both a saturated or wet surface dry condition (SSD) and an oven dried condition (OD). The purpose of preparing compacts for both of these conditions was to determine, by the percentage of evaporable water at 220 F (105 C), an approximation of the final porosity in the compacted material upon completion of the desired curing period. From the duplication of the series in this way, the data determined from the oven dried compacts could be translated to the strengths achieved in the saturated surface dry condition (SSD). As will be shown later, the process of oven drying has a significant effect in producing a higher strength material due to restrained shrinkage stresses which develop in the dried compact and is therefore uncharacteristic of the strength which actually exists in a moist compact.

Unforeseen at the initial outset of the laboratory program design phase were the complications associated with the high pressure compaction of two very different powder materials. Fig. 6 and 7 , typical cement and fly ash material used for this investigation, shows

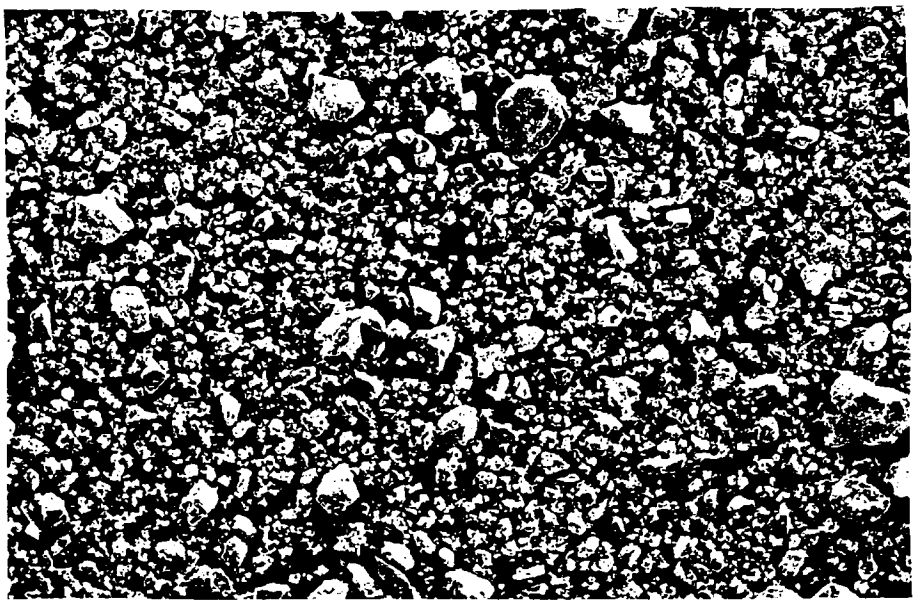

Fig. 6 - Typical Type III portland cement material used in the compacts magnified 300 times

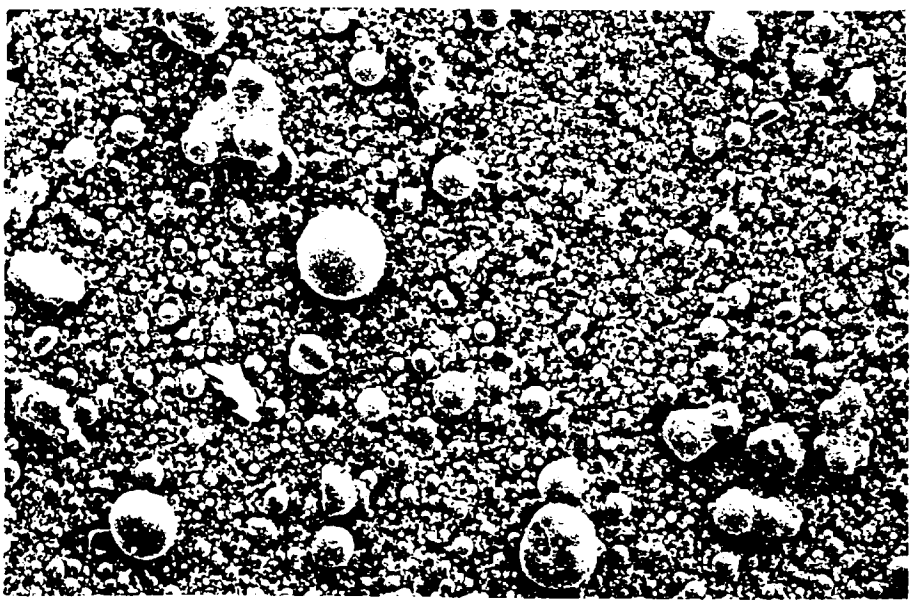

Fig. 7 - Typcial Class "C" fly ash material used in compacts magnified 300 times

the variations in size and shape between the two types of material. It is interesting to note the rough, angular, irregular shaped particles of the cement material in comparison to the smoother, more spherical particles of fly ash material.

After some laboratory experimentation it was found that the raw fly ash used contained, in relation to the cement powder, some extremely large particles which tended to disrupt the proper high pressure compaction of the compacts. It was decided to eliminate the larger spherical fly ash particles which were not compatible with the angular crushed cement particles by using only fly ash which passed a No. 100 wire mesh sieve (i.e. < 150 microns).

In addition to the larger fly ash particles, this sieving process also eliminated virtually all visible unburned carbon particles from inclusion in the compacts. Because of this a more compatible gradation of fly ash was utilized and the end product, the prepared compacts, were more physically stable. The compatibility of the fly ash with the cement in this high pressure compaction process was not the intent of this work and for this reason will only be briefly mentioned. Even though a number of series were prepared utilizing the coarser raw fly ash material, the remainder of this report will deal only with data obtained from the compacts prepared from the sieved fly ash material.

From the various considerations discussed, the laboratory program shown in Table 1 was developed and utilized throughout the project. This table shows the compacts which were prepared for the various parameter combinations which existed. The unsieved fly ash material

Table 1 - Target calculated initial porosity percentages for the laboratory program

Curing Period (days)	Percent Fly Ash Replacement For Cement			
	0	10	20	30
3	20,23,26,29,32			
7	20,23,26,29,32	20,26,32 S	20,26,32 U,S	20,26,32 S
28	20,23,26,29,32	20,26,32 S	20,26,32 U,S	20,26,32 S
90	20,23,26,29,32	20,23,26,29,32 U,S	20,23,26,29,32 U,S	20,23,26,29,32 U,S

Legend:
1. S denotes compacts were prepared with sieved fly ash material passing a No. 100 sieve.
2. U denotes compacts were prepared with unsieved fly ash material.

Note: All parameter combinations were prepared for testing in the Saturated Surface Dry and Oven Dried conditions.

was utilized only in compacts cured for 90 days at all percentages of fly ash replacement and also in compacts containing 20 percent fly ash and cured for the 7 and 28 day durations. The results of strength tests on these compacts are not included in this report. The remainder of the compacts prepared according to this table are included in the data presented and analyzed later in this report.

For control purposes and ease in terminology throughout the project, the term "series" was used to define set of prepared compacts having the same parameter values as defined in Table 1. Each "series" consists of a minimum of six individual compacts all having the same percentage of fly ash replacement for cement, same target initial porosity and same curing duration.

A total of 74 series were prepared for testing in the saturated surface dry condition condition and an additional 74 series for the oven dried condition. On the average, eight compacts were prepared for each series, which totaled the preparation of approximately 1200 compacts for curing and subsequent compressive strength determination.

To keep accurate and efficient records of all the compacts prepared, the compact and series numbering system shown in Fig. 8 was used throughout the project. Note that an alphabetic symbol (B, C, D, or E) was used to denote curing duration; an upper case letter denotes a compact tested in a wet surface dry condition and a lower case letter denotes a compact tested in an oven dried condition. For the parameters of calculated initial porosity and percentage fly ash replacement of the cement material, numerical symbols as denoted in

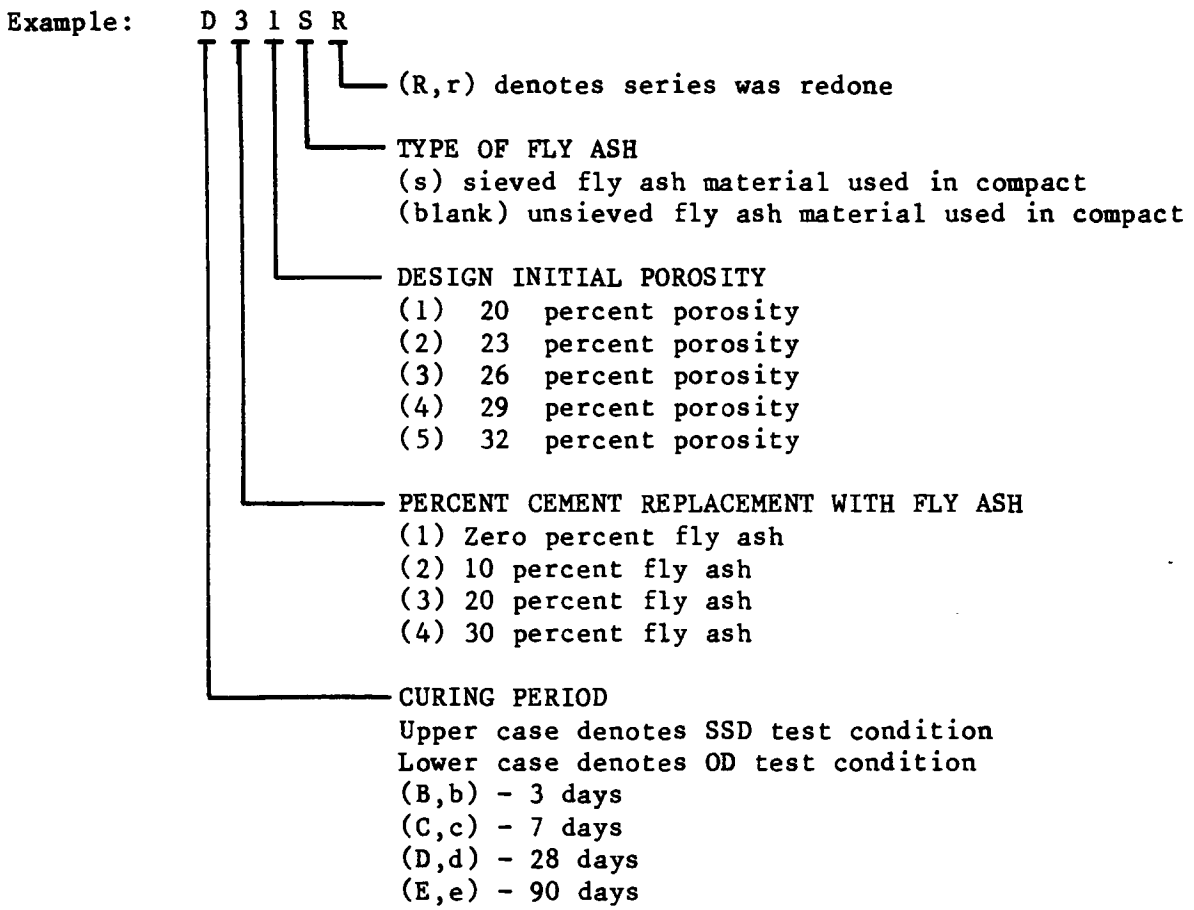

Fig. 8 - Compact series numbering system

the figure were utilized. The letter "S" was used to denote when the sieved fly ash material was used in place of the unsieved fly ash. If no "S" is present then either the unsieved fly ash material was used or a cement only condition existed. The letter "R" was added later in the laboratory investigation when it became evident that remakes of some of the compacts would be necessary in an effort to collect usable data. In addition to the symbols shown, each compact of a particular series was assigned a unique alpha-numeric symbol to distinguish it among the individual compacts in a particular series.

Performance of the Laboratory Program

The first major step in the performance of the laboratory investigation was to determine the relationship between the amount of powder used, required production pressure, and the porosity which was produced by that amount of powder. For this determination, ten series of three to eight compacts each were prepared from various amounts of anhydrous cement powder. The initial porosities were then calculated using the bulk volume, weight, and specific gravity of the cement and a linear regression analysis performed to relate the amount of powder added to the die to the porosity obtained upon full compaction. Full compaction is defined as the compaction effort at the time the ram head comes in contact with the spacer piece.

The resulting linear regression equation calculated was:

$$P_i = - 17.8013\ W_o + 71.2672 \qquad (4)$$

where P_i is the calculated initial porosity, and

W_o is the weight of powder placed in the die for compaction.

The r^2 or coefficient of determination[36] for this equation is 0.994. Further discussion of the development of this equation is contained in Appendix C.

Due to this excellent relationship, it was simply a matter of solving the equation to determine the amount of powder to be used in producing a desired initial porosity. It is important to note that W_o is the weight of powder placed in the die before the compaction process and not the weight of the compact upon preparation. This method of powder weight determination is advantageous in that the minor loss of powder between the ram and the die walls during the compaction process is taken into account.

The next major step, using the weight of anhydrous cement and resulting porosity relationship as control, was to determine the powder weights needed to produce the desired porosity at each level of fly ash replacement for the cement. This was accomplished through the determination of a factor which accounted for the specific gravity of the fly ash and the percentage of fly ash replacing cement in a particular compact. Once this factor was calculated, the needed powder weight to produce a compact of desired porosity with a certain amount of fly ash replacement was determined by multiplying the anhydrous powder weight of cement needed to produce a certain porosity by the appropriate factor. By using this method, a significant

amount of laboratory time was saved as the determination of the relationship between powder weight for each percentage of fly ash replacement and resulting porosity was avoided. A complete discussion of this procedure is contained in Appendix C.

Once the weight of powder to produce a desired initial porosity was determined for each percentage of fly ash, the actual performance of the laboratory program was very repetitious in nature. Fig. 9 shows the essential steps which were followed in the production of the compacts. Keep in mind that this is only a general overview of the procedures which were followed. A more in depth description of the actual step-by-step procedures for the preparation, curing, and testing of the compacts may be found in Appendix B.

As shown in Fig. 9, the laboratory procedure followed a sequence of events which consisted, in simplest terms, of preparation of the powder to be compacted, production of the compacts, weighing and measuring of the freshly prepared compacts and subsequent curing for the desired duration. Upon completion of curing, the compacts were weighed and measured in a saturated surface dry condition. Depending on the initial intent of the individual series, the compacts were then tested for compressive strength in a saturated surface dry condition (SSD) or placed in a convection oven at 220 F (105 C) for subsequent determination of the final porosity present in the hydrated compacts. Following the oven drying process, these compacts were also tested for compressive strength.

The last phase of the laboratory investigation involved the data preparation and analysis of all the various dimensions and weights

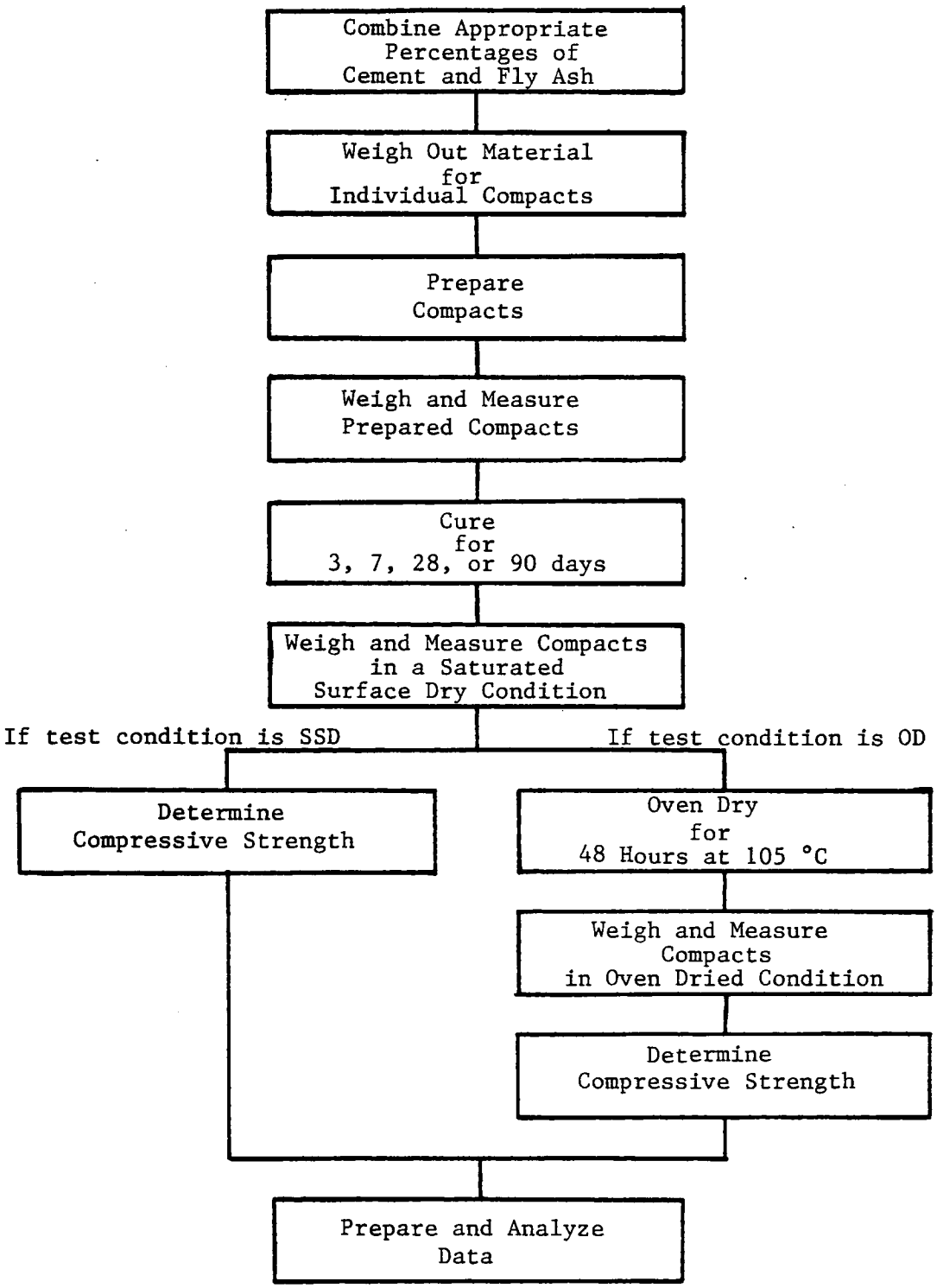

Fig. 9 - Flow chart of laboratory procedures

obtained during the actual performance.

For each of the individual compacts prepared, from the initial weight and dimensions the initial porosity was determined. The equation used for the calculation of the intial porosity was:

$$P_i = 1 - ((W_1/V_o)((\%C/SG_c) + (\%FA/SG_{fa}))) \quad (5)$$

where P_i is the calculated initial porosity,

W_1 is the initial weight of the prepared compact,

V_o is the volume of the prepared compact,

%C is the percentage by weight of cement in the compact,

%FA is the percentage by weight of fly ash in the compact,

SG_c is the specific gravity of the cement, 3.13 ,

SG_{fa} is the specific gravity of the fly ash, 2.71 for the unsieved fly ash material and 2.75 for the sieved fly ash material. The derivation of this equation is produced in Appendix D.

Once the calculated initial porosity was determined for each individual compact of a certain series, an average calculated initial porosity was determined for that series using the values of the individual compacts. A standard deviation value and coefficient of variation were also determined for each series. The same procedure was also followed for the various other items of data contained in Appendix E. In this way the values for each unique series are an average of the values for the six to eight individual compacts which compose the series. All further data analysis which is presented in

the following chapters is based on these average values for each individual series.

CHAPTER IV

PRESENTATION AND DISCUSSION OF LABORATORY RESULTS

Introduction

The purpose of the following discussion is to present the data collected and observations made in the performance of the laboratory portion of the study. The figures contained in this Chapter were developed from the data contained in Appendix E.

The discussion in this Chapter will be separated into two categories. Since the die equipment used for this project was a special design and is expected to be used in further studies concerning the high pressure compaction of cement materials, it is appropriate to discuss the various relationships which resulted from this study concerning initial weight of powder material, production pressure and resulting calculated initial porosity. The second portion of the discussion will pertain to the results obtained concerning the effects of the main parameters, namely percentage fly ash replacement for cement, calculated initial porosity and the curing duration, on the strength of the prepared compacts.

Much of the data analysis contained in this section utilized linear regression analysis. However, Appendix F contains the pertinent data for each such analysis performed and is sorted by the figure for which the equation applies. As the equations were generally not determined for predictive purposes, for the figures in this section only the coefficient of determination (r^2) for each

relationship are included on the figure.

Results of Die Operation

Fig. 10 shows the relationship between the weight of the prepared compacts and the calculated initial porosity achieved for the various percentages of fly ash replacement for cement. Note the distinct delineation between the various percentages of fly ash. For all practical purposes, the weight values listed in this figure may be used for the approximation of the amount of powder material needed to produce a certain initial porosity.

This figure was developed by performing a linear regression analysis on all the series prepared for this study. For example, the relationship between prepared compact weight and calculated initial porosity for 20 percent fly ash replacement was developed by performing a linear regression analysis using the prepared compact weight and the calculated initial porosity for each series prepared containing 20 percent fly ash. The same procedure was used to determine the relationships between prepared compact weight and calculated initial porosity for zero, 10, and 30 percent fly ash amounts. Judging from the coefficients of determination listed on the figure, the linear relationship determined between prepared compact weight and calculated initial porosity is excellent.

Comparing the values for prepared compact weights with the weight of powder initially placed in the die to achieve a desired porosity as discussed in Appendices B and C it appears that the initial prediction equation (Eq. 4 in Chapter III and Eq. 2 in Appendix C) for the

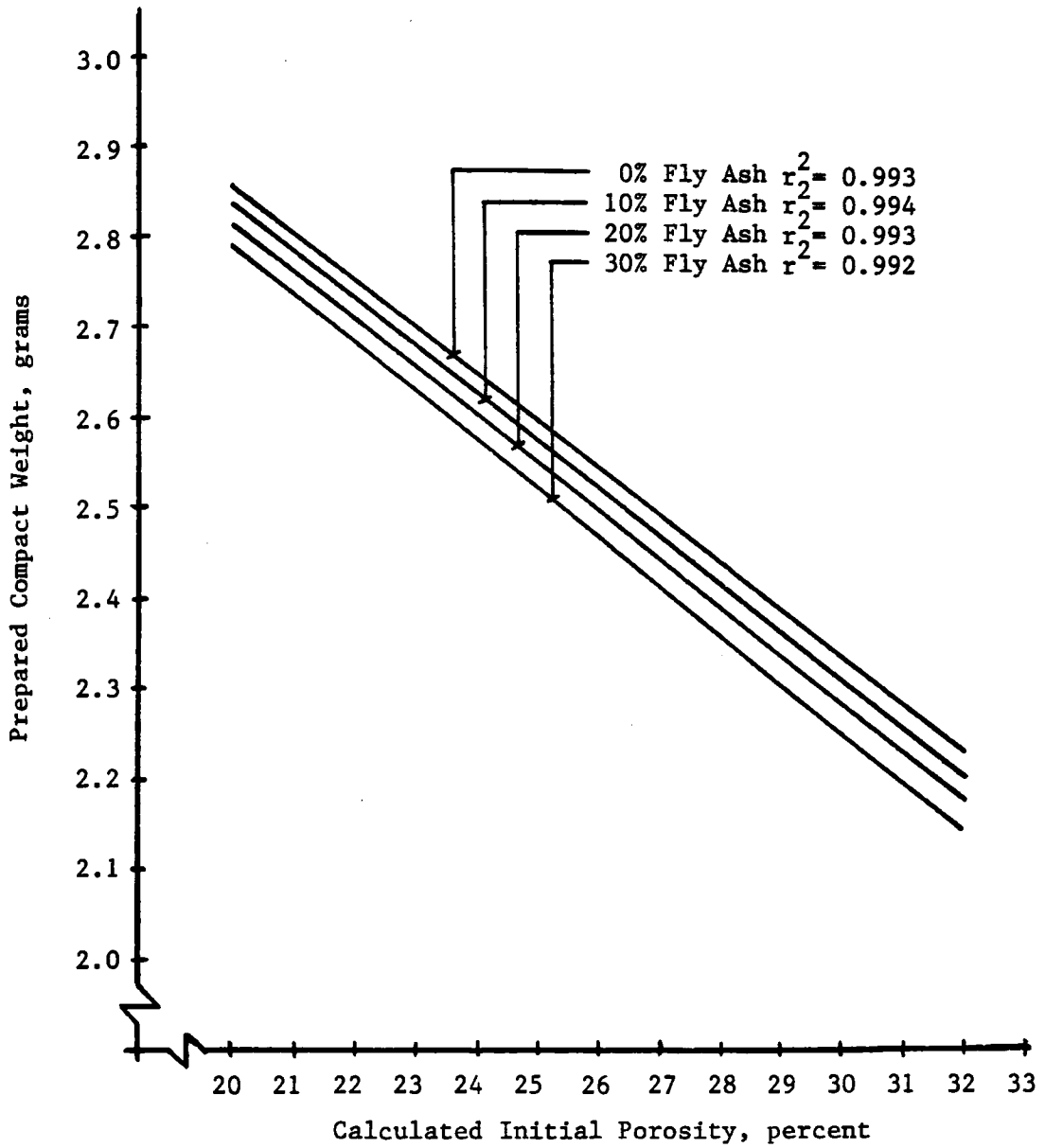

Fig.10- Prepared compact weight vs. calculated initial porosity relationship for zero, ten, twenty and thirty percent fly ash replacement

cement only (zero percent fly ash) was very adequate in determining the amount of powder necessary to produce a certain initial porosity. For the various percentages of fly ash (10,20,20 percent) however, the initial powder weights used appear to be less than the amount actually needed to produce a target initial porosity.

This can be observed by entering Fig. 10 with the weight of powder material needed to produce a desired initial porosity at a certain percentage of fly ash replacement, as taken from Table 3, and reading the corresponding calculated initial porosity which would be obtained. This signifies that the factors which were used, as discussed in Appendix C, to determine the amount of material containing fly ash based on the amount of cement material only were slightly lower than needed. From further consideration, this means that the specific gravity of the fly ash material is perhaps greater than the 2.75 value determined in the laboratory and used throughout the project. This in turn accounts for the increasing deviation between the desired porosity to the actual initial porosities obtained as the percentage of fly ash increases. The amount of this deviation however is not significant enough to have caused any problems in the actual performance of the investigation.

Fig. 11 shows the relationship between the weight of the prepared compacts and the production pressure to produce a compact at that weight. As one would expect from the discussion on operation of the die and the full compaction concept, as the weight of the compact increases the production pressure also increases. Note that the

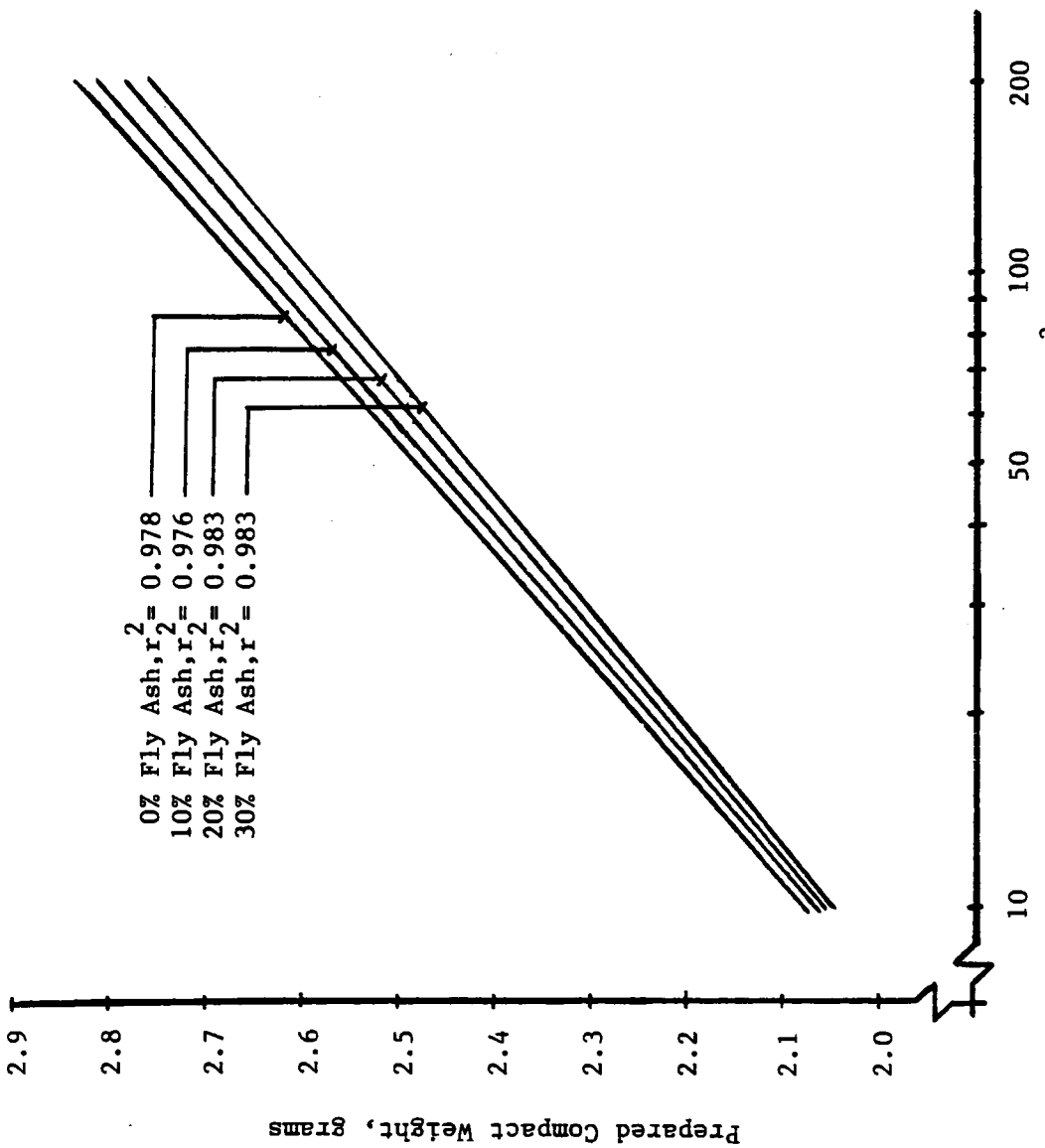

Fig. 11 - Prepared compact weight vs. production pressure relationship for zero, ten, twenty, and thirty percent fly ash replacement (Note: 1 psi = 0.006895 MPa)

relationship is linear when plotted semi-logarithmicly.

As with the prepared compact weight and calculated initial porosity relationship, this figure was also prepared by using the prepared compact weight and the associated production pressure for each series prepared with the same percentage of fly ash in a linear regression analysis. For this analysis however, the base 10 logarithm of the production pressure was used in order to produce a linear relationship. The accompanying coefficients of determination signify an excellent relationship between the two.

Again, as with the weight - porosity relationship of Fig. 10, a distinct delineation between the various percentages of fly ash can be observed. For a particular production pressure, as the percentage of fly ash decreases in the compacts, the weight of the powder needed and the resulting weight of the prepared compacts will increase. This is due to the lower specific gravity fly ash material.

There also appears to be a much greater difference between the weight of the prepared compacts containing zero percent fly ash and 30 percent fly ash at the higher production pressures than at the lower production pressures. The reason for this is unclear. One possible reason is that at the higher production pressures more powder material is needed and the effects of an inaccurate specific gravity value for the fly ash may be compounded. Another possbile reason is, due to the contrast in particle shape and texture of the two materials, the amount of compaction and porosity reduction varies. Also possible may be increased particle surface area in the compact caused by the crushing of the fly ash particles in the compact at extremely high

loading pressures. Should this be the case then the formation of a greater number of smaller particles resulted in larger porosities and thus less powder weight to achieve a full compaction condition as defined in Chapter III.

From the excellent relationships concerning prepared compact weight, calculated initial porosity, and production pressure it is appropriate to consider the calculated initial porosity and the base 10 logarithm of production pressure as being synonymous in the analysis of data. Fig. 12 was developed to show that calculated intitial porosity is inversely proportional to the log of the production pressure. Note the excellent correlation (r^2= 0.991) even though production pressure and resulting initial porosity data for all percentages of fly ash was used to determine this relationship. The synonymous use of the terms production pressure and porosity throughout the remainder of this report is an underlying point.

Results Pertaining to the Initial Parameters

This section presents the test results pertaining to the three main parameters, calculated initial porosity, various percentages of fly ash replacement for cement, and effects of various curing durations, on the compressive strength of the prepared compacts.

Since the compacts prepared for the oven dried and wet surface dry testing conditions had the exact same parameter values, for the purposes of this work the oven dried compacts were prepared solely to determine a measure of the final porosity present in the compacts upon

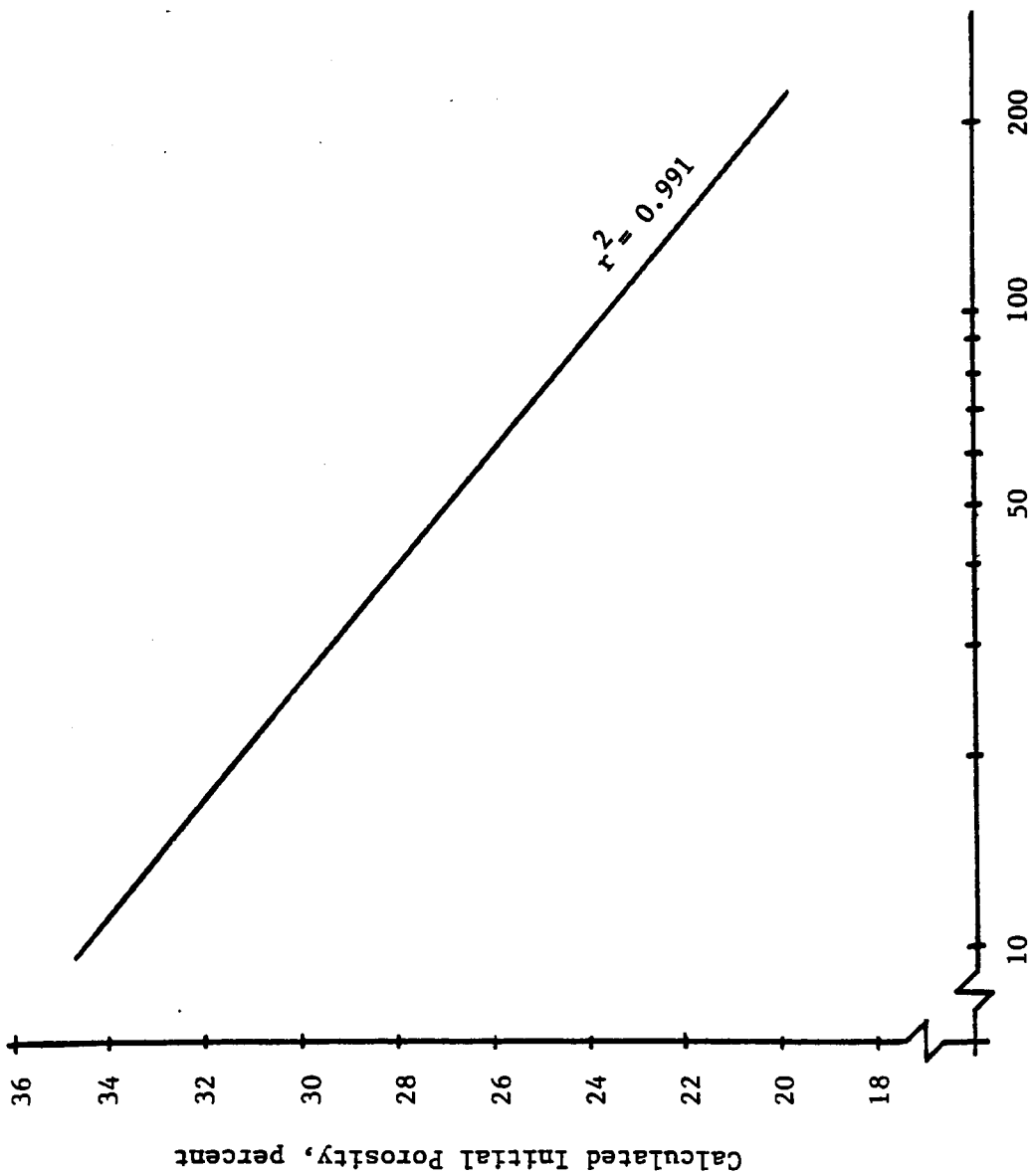

Fig. 12 - Normalized calculated initial porosity vs. production pressure relationship (Note: 1 psi = 0.006895 MPa)

completion of curing. From this final porosity determination of the oven dried compacts, a regression analysis was performed to relate the final porosity to the initial porosity of the prepared compacts. From the equations developed, the final porosity of the compacts tested in the wet surface dry conditon were estimated and used for data analysis purposes.

Since the final porosity condition is dependent upon the initial porosity condition and the synonomous production pressure, the relationship between final porosity and production pressure was analyzed. Fig. 13 shows the results of the linear regression analysis. The best correlations were achieved by considering each percentage of fly ash replacement separately. For a given initial porosity, as the percentage of fly ash is increased, the final porosity also increased. This indicates the formation of less hydration product and resulting decrease in final porosity as the percentage of fly ash increases.

It also appears from the general relationship of the lines representing the various percentages of fly ash that effects of fly ash on the final porosity become less as the production pressure is decreased or the initial porosity is increased. This indicates that at the higher initial porosity conditions, regardless of the percentage of fly ash, porosity is reduced the same amount by the formation of hydration products. At the lower initial porosity conditions, corresponding to higher production pressure, the formation of hydration product and the resulting final porosities are the lowest when no fly ash is used. One possible explanation may be that the low

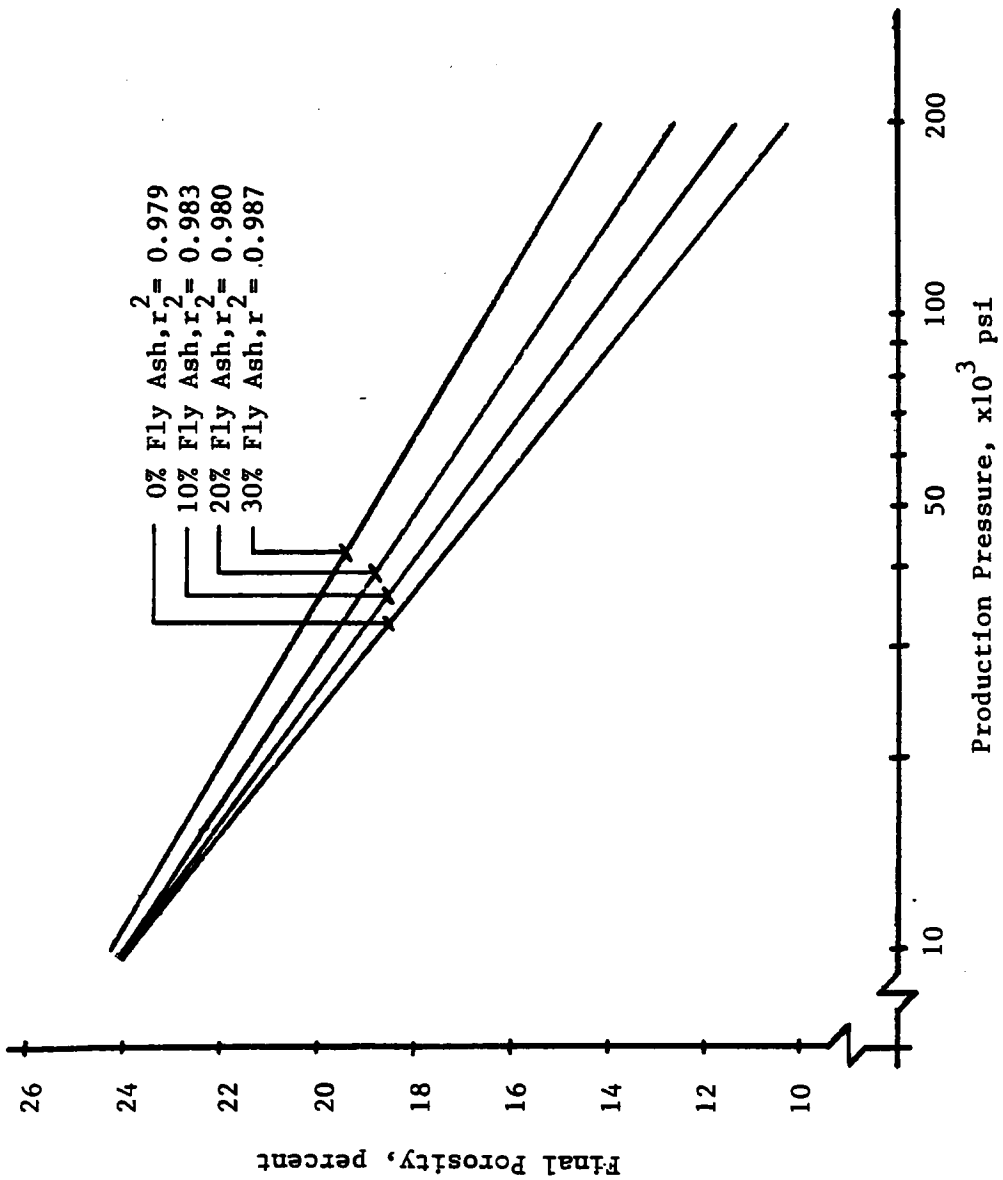

Fig. 13 - Final porosity vs. production pressure relationship for zero, ten, twenty, and thirty percent fly ash replacement (Note: 1 psi = 0.006895 MPa)

initial porosity conditions increasingly inhibit the formation of hydration products. Another key point is that with increasing amounts of fly ash, less cement is present. This condition in turn reduces the amount of hydration product and calcium hydroxide that is formed as a result of the hydration process.

This could further be explained as a direct relationship to the amount of available calcium hydroxide available for pozzolanic reaction. As discussed in Chapter II, calcium hydroxide will only form in available free space. It is believed that the low porosity condition may negatively affect both hydration and pozzolanic reactions as the available space for the reactions to occur is reduced. In this way two of the important ingredients necessary for a pozzolanic reaction are effectively reduced in the low porosity condition. This could explain the increasing final porosity contents as the amount of fly ash increases at the low porosity conditions.

From the various measurements taken throughout the laboratory investigation density determinations can be made at the three distinct stages of the testing process, namely the initial density of the compacts before hydration, a wet surface dry density as the compacts are removed from the curing solution and an oven dried density after oven drying for 48 hours. From the comparisons of these densities some insight into the amount of hydration product formed can be developed to further support this point of varying degrees of hydration product formation.

In observing Fig. 14, 15, 16 and 17 it is necessary to point out

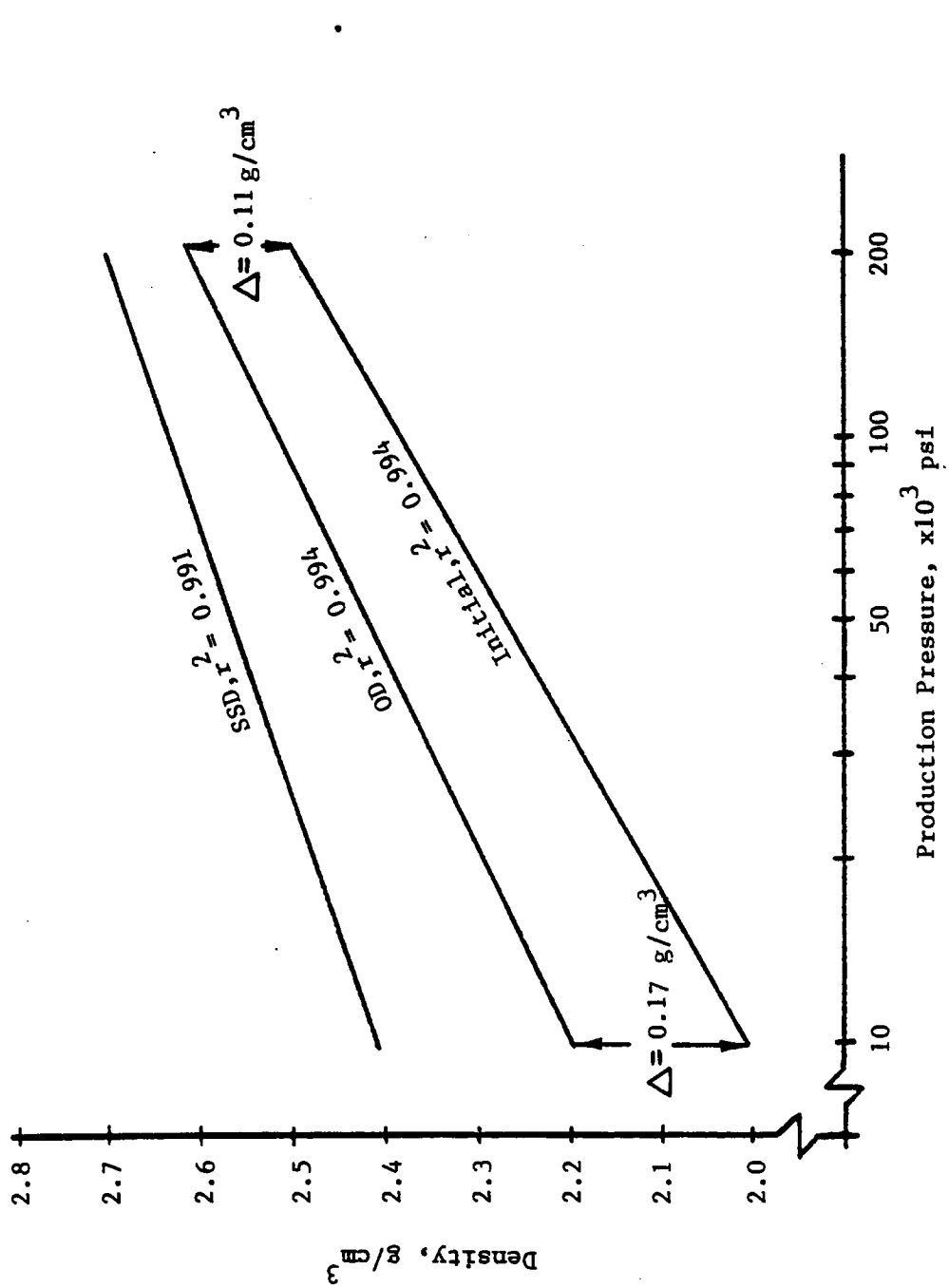

Fig. 14- Density vs. production pressure relationship for 100 percent portland cement compacts
(Note: 1 psi = 0.006895 MPa)

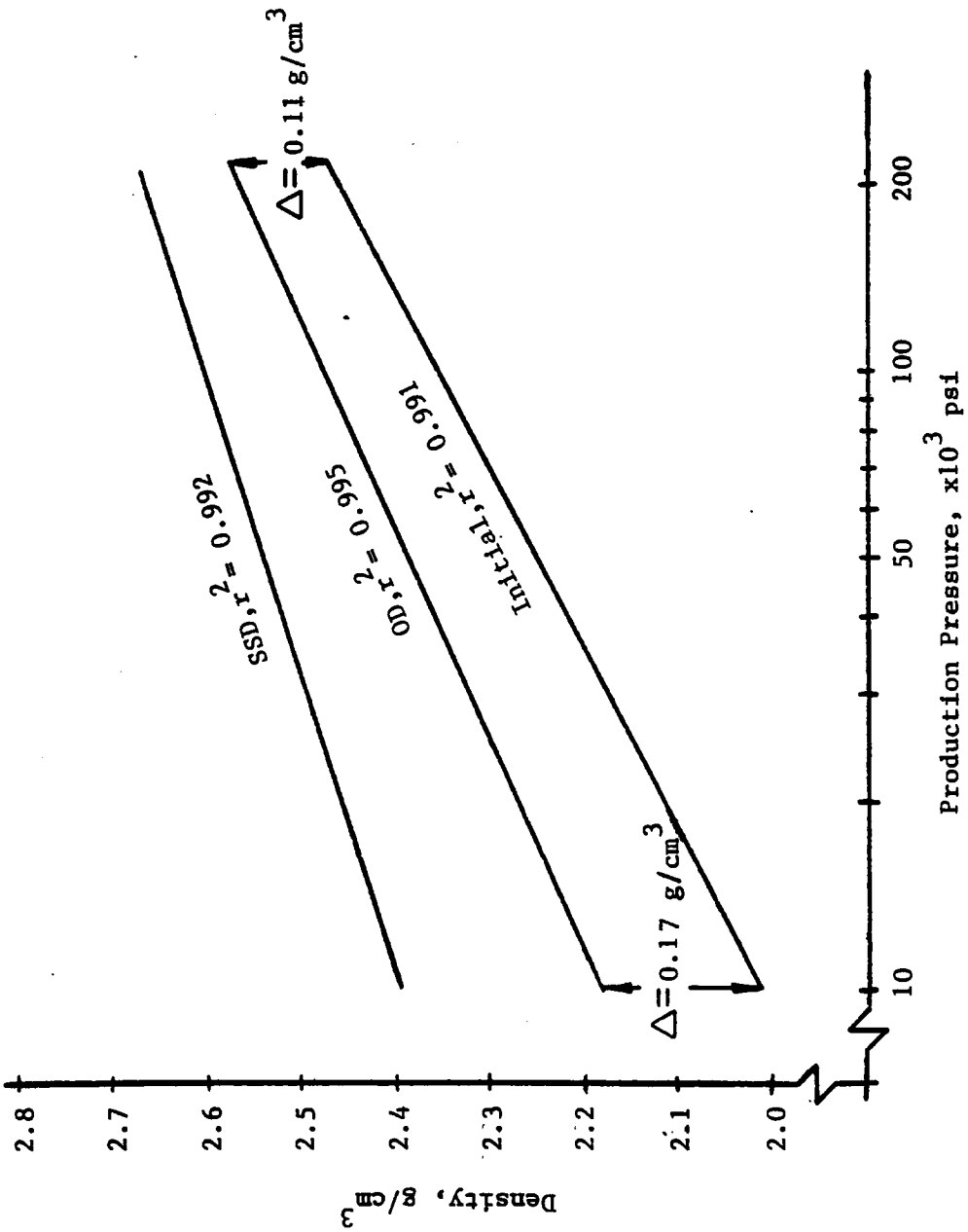

Fig.15 - Density vs. production pressure relationship for compacts containing ten percent fly ash (Note: 1 psi = 0.006895 MPa)

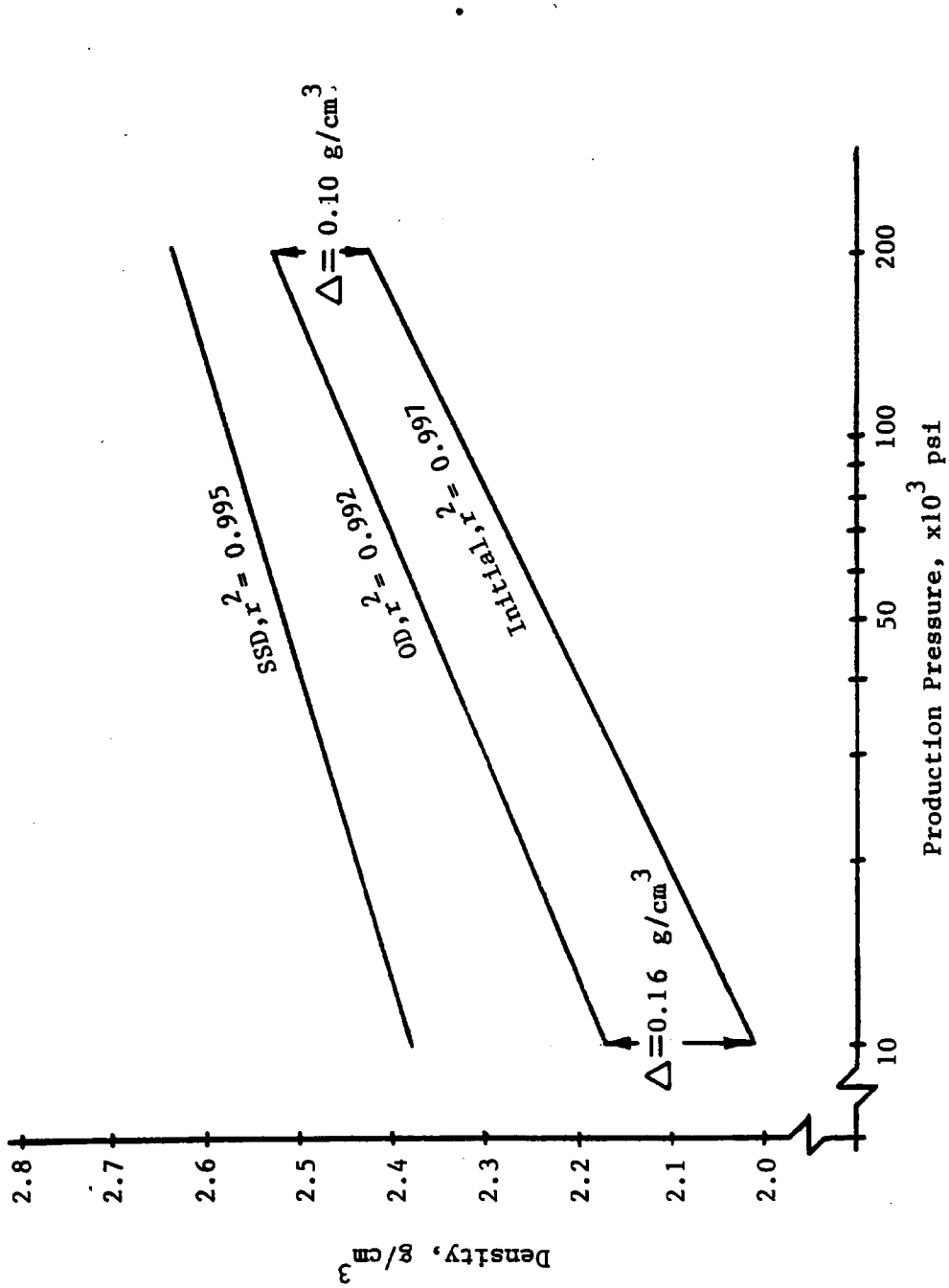

Fig. 16 - Density vs. production pressure relationship for compacts containing twenty percent fly ash (Note: 1 psi = 0.006895 MPa)

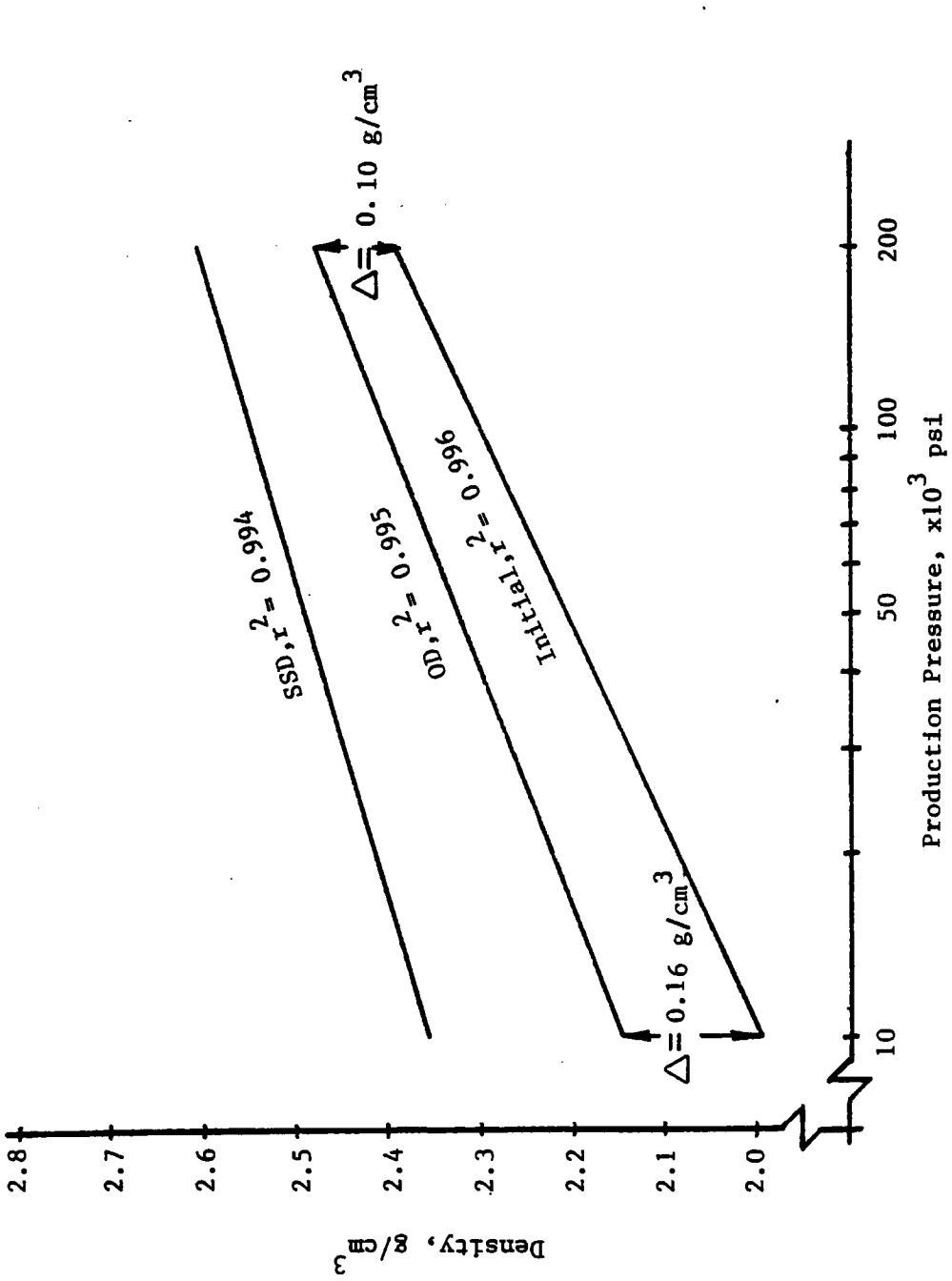

Fig. 17 - Density vs. production pressure relationship for compacts containing thirty percent fly ash (Note: 1 psi = 0.006895 MPa)

the relationships between the various densities. The initial density, as mentioned, is the density of the compacts upon preparation and no hydration. As one would expect, as the production pressure increases the density of the compacts increases due to the decrease in porosity. The wet surface dry density is the density of the compact upon removal from the curing solution at the completion of the appropriate curing duration and includes the weight of the free water contained in the pores of the compact. The oven dried density is the density of the compact upon completion of oven drying and elimination of essentially all free water.

By comparing the density of the oven dried compact with the initial density of the compacts, the difference in the two is a measure of the amount of hydration product which has formed. Of course the oven dried compact will still contain a certain amount of combined water in various stages which will be a certain percentage of the oven dried density. If this quantity of combined water is considered to be a constant percentage of the hydration product formed, then the general trends should be valid. In the figures that follow, for the purpose of making the point that varying degrees of hydration exist at the high and low porosity conditions, the various densities at the different curing durations were combined as to percentage of fly ash. In this respect, the relationship which are shown are considered "average".

Fig. 14 shows the density - production pressure relationships for the cement only (zero percent fly ash) compacts in the initial, oven dry (OD), and saturated surface dry (SSD) conditions. The

relationships between the various types of densities is obvious. Upon closer examination of the relationship between the oven dried density and the initial density, it appears that the difference in density (0.17 g/cm^3) from the initial to oven dried condition at the lowest production pressure or higher initial porosity condition is greater than the difference in density (0.11 g/cm^3) at the higher production pressure or lower initial porosity condition. This signifies the formation of more hydration products at the higher porosity conditions than at the lower porosity conditions.

Fig. 15, 16, and 17 show that similar relationships exist for the various percentages of fly ash replaced for cement. The general trends are the same as that of the cement only. Of interest is the fact that as the percentage of fly ash in the compacts increases, the differences in the density changes at the lowest and highest production pressures decreases. This is believed to signify the formation of less hydration products as the percentage of fly ash is increased, coupled with the formation of less hydration products as the production pressure is increased.

Since the density of the compacts is a function of the level of porosity they contain, examination of the change in porosity from the initial to the final condition should tend to support the statement that less hydration product formation occurred as the production pressure increased or the initial porosity was lowered. This is exactly the case as Fig. 18 shows.

Fig. 18 is a normalized representation of all the porosity -

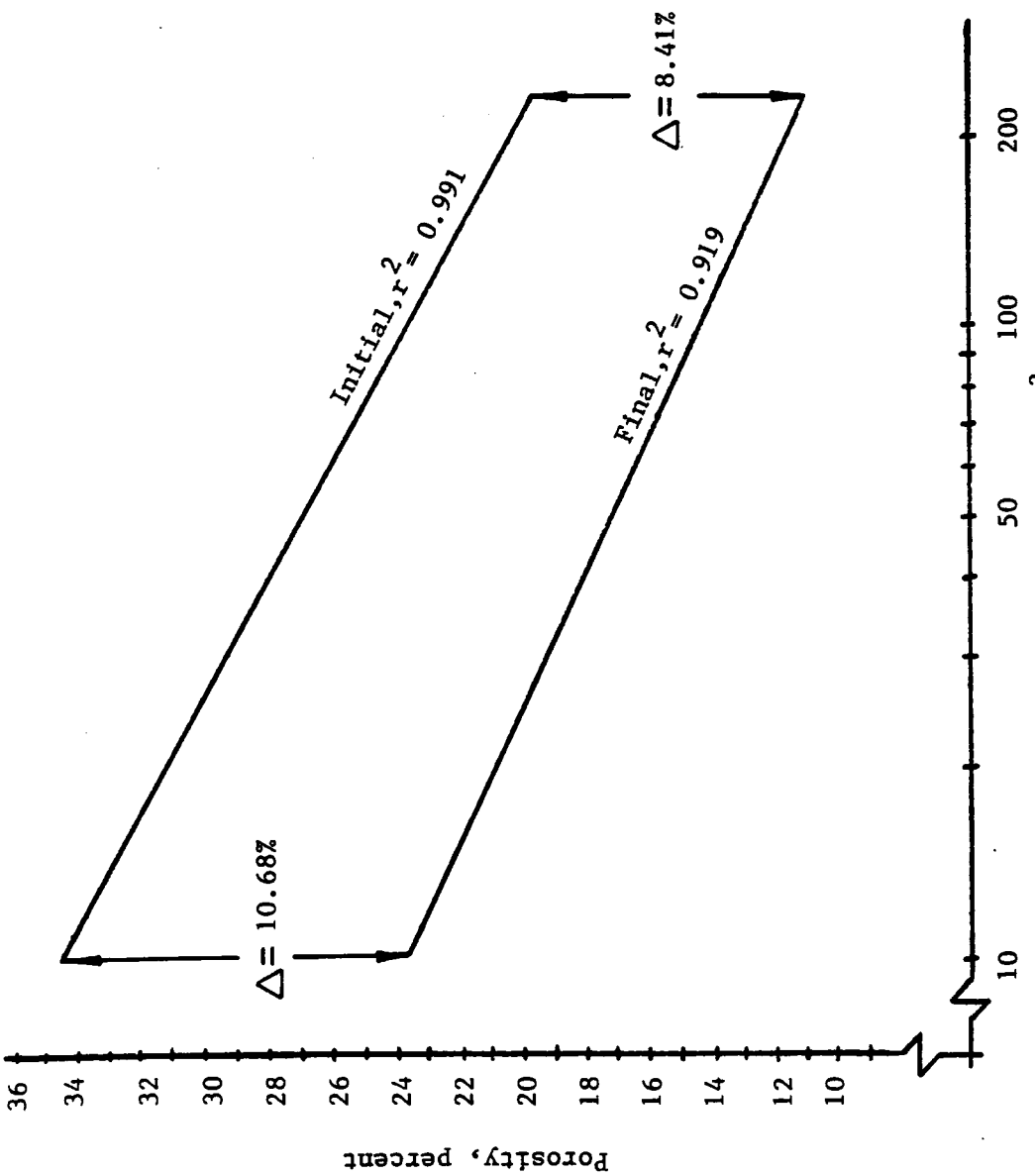

Fig. 18 - Normalized final and initial porosity vs. production pressure relationships
(Note: 1 psi = 0.006895 MPa)

production pressure data obtained during the investigation. The initial porosity line is the same as shown in Fig. 12. To determine the change in porosity from the initial to final conditions, the final porosity line was added for comparison purposes.

For the miniature compacts prepared at the highest production pressure, regardless of the duration of curing and the percentage of fly ash replacement for cement, the total average change in porosity was 8.41 percent. The compacts prepared at the lowest production pressure had an average change in porosity of 10.68 percent. This supports the statement that increased hydration product formation occurs at conditions of greater porosity resulting in a greater percentage reduction in porosity.

From the figure it is also interesting to note the relationship between the initial reduction of porosity due to the high pressure compaction process and the subsequent reduction in porosity due to the hydration process. At the highest production pressure, the resulting change in porosity of 8.41 percent from the initial to the final conditions was a 41.43 percent reduction in the amount of the initial porosity. At the lowest production pressure, the resulting change in porosity of 10.68 percent resulted in only a 30.96 percent reduction in the amount of initial porosity. In this respect then, the formation of hydration products in compacts prepared at higher production pressures provide considerably more influence to porosity reduction than hydration products formed in compacts prepared at lower production pressures. This is in aggreement with the principle of increased specific binding capacity as discussed in Chapter II.

As a summary to this discussion, Fig. 19 provides some interesting insight into these relationships. In general, the relationship between production pressure, reduction in porosity and the percentage of fly ash replacement is such that the formation of hydration products increases as the production pressure and percentage of fly ash replacement decreases. On a percentage basis however, the hydration products formed in compacts produced at higher production pressures have a greater influence on porosity reduction than compacts prepared at lower production pressures.

At the zero percent fly ash level, a wide variation in porosity reduction occurs depending on the production pressure and resulting initial porosity. However at the 30 percent fly ash level, the percent reduction in porosity is relatively insensitive to the production pressures. This shows the influence of greater hydration product formation in the compacts containing no fly ash and the associated production pressure and initial porosity on the degree of reduction in total compact porosity which occurs upon curing. Again the relative increasing insensitivity of reduction in porosity as the percentage of fly ash increases is believed due in large part to the fact that less cement is present for the hydration reaction.

Having seen the influence of the production pressure on the density and associated porosity of the compacts, it is appropriate to discuss the influence of the production pressure on the resulting compressive strengths. Since production pressure and initial porosity are synonymous, this discussion will essentially relate the

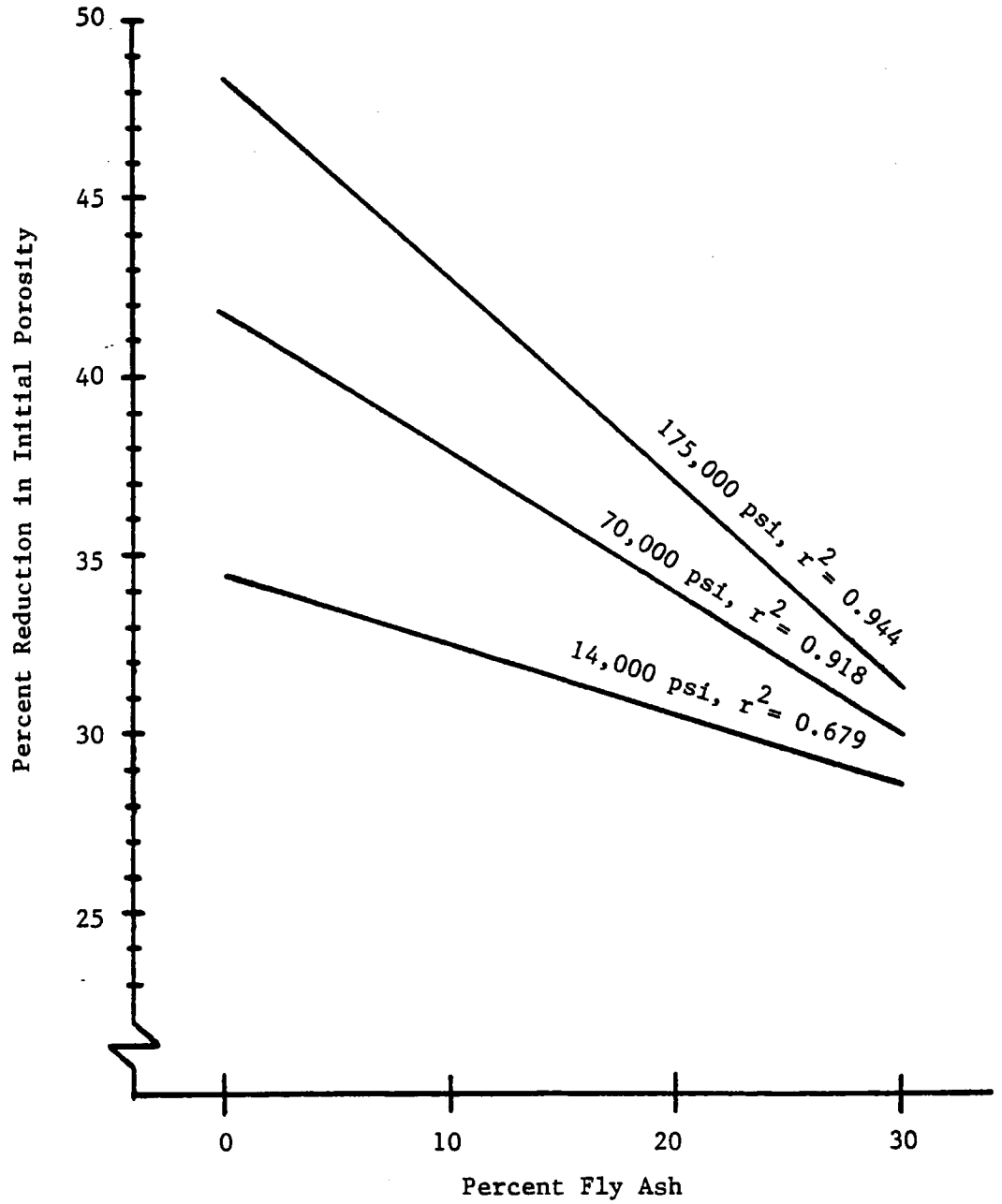

Fig.19 - Percent reduction in porosity vs. percentage fly ash replacement relationship at 14,000, 70,000, and 175,000 psi production pressures (Note: 1 psi = 0.006895 MPa)

compressive strengths obtained to the initial porosity conditions.

Fig. 20 shows the compressive strength results in relation to production pressure for the zero percent fly ash compacts. Generally as the production pressure increases the compressive strength at all ages also increases. The influence of curing duration on the resulting compressive strength is also clearly evident. It is interesting to note the significant increase in strength at all production pressures from the 3 to 7 day curing period and the smaller increase in strength which occurred from 7 to 28 days. Unfortunately due to improper testing procedures strength data for cement only compacts cured for 90 days is not available.

Also of importance from this particular figure is the magnitude of the strength increases at the various production pressures. This is seen by observing the smaller difference in magnitude of strength gain at the highest production pressure in relation to the greater amount of strength gain at the lower production pressures. This leads one to believe that for higher production pressures, greater strengths at early ages are developed. However, due to the formation of greater amounts of hydration products in the compacts of higher porosity, the strengths of the compacts produced at the lowest production pressures approach the strengths of the compacts prepared at the higher production pressure as curing duration increases. This trend in strength increase at lower production pressures again supports the conclusions drawn from the earlier figures that a greater amount of hydration occurs in compacts with greater levels of porosity.

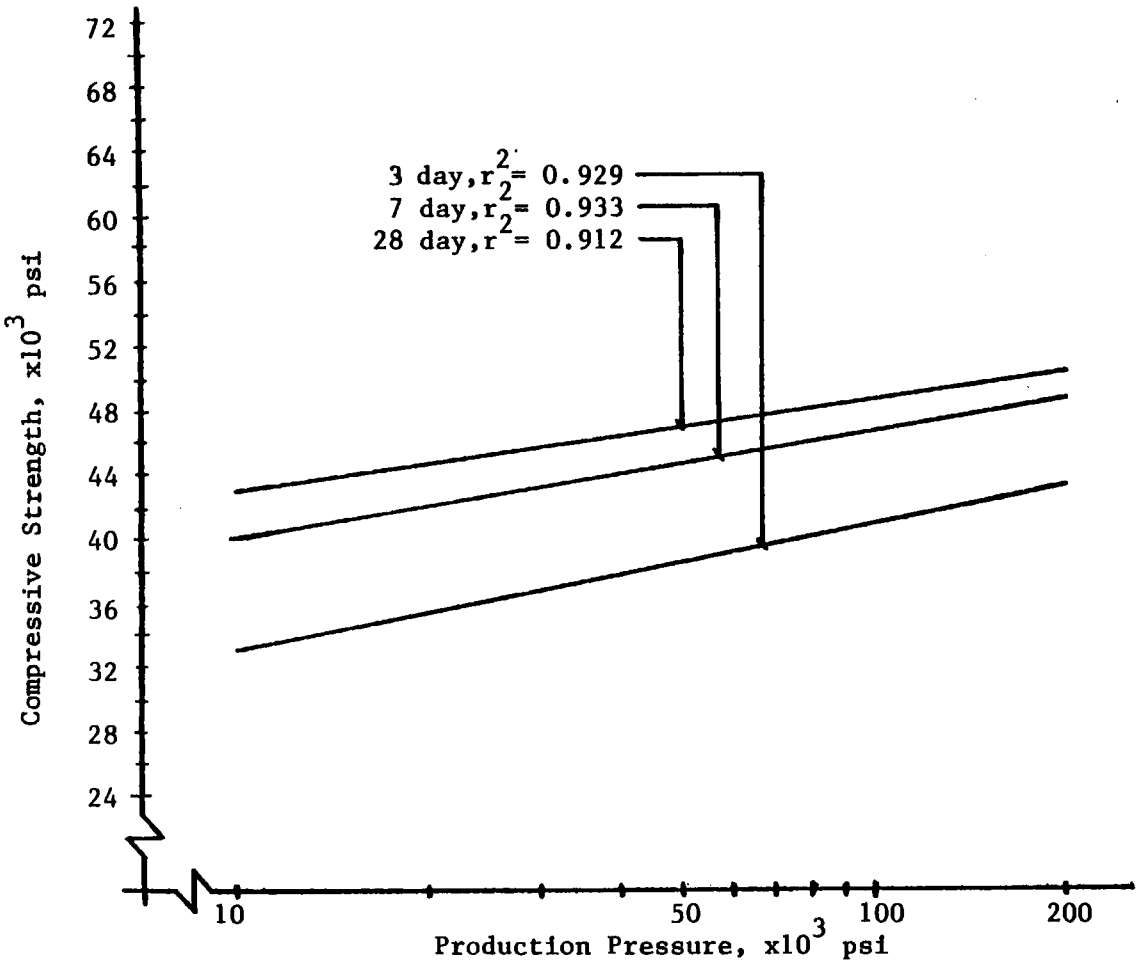

Fig. 20 - Compressive strength vs. production pressure relationship for 100 percent portland cement compacts at three, seven, and twenty-eight days of age (Note: 1 psi = 0.006895 MPa)

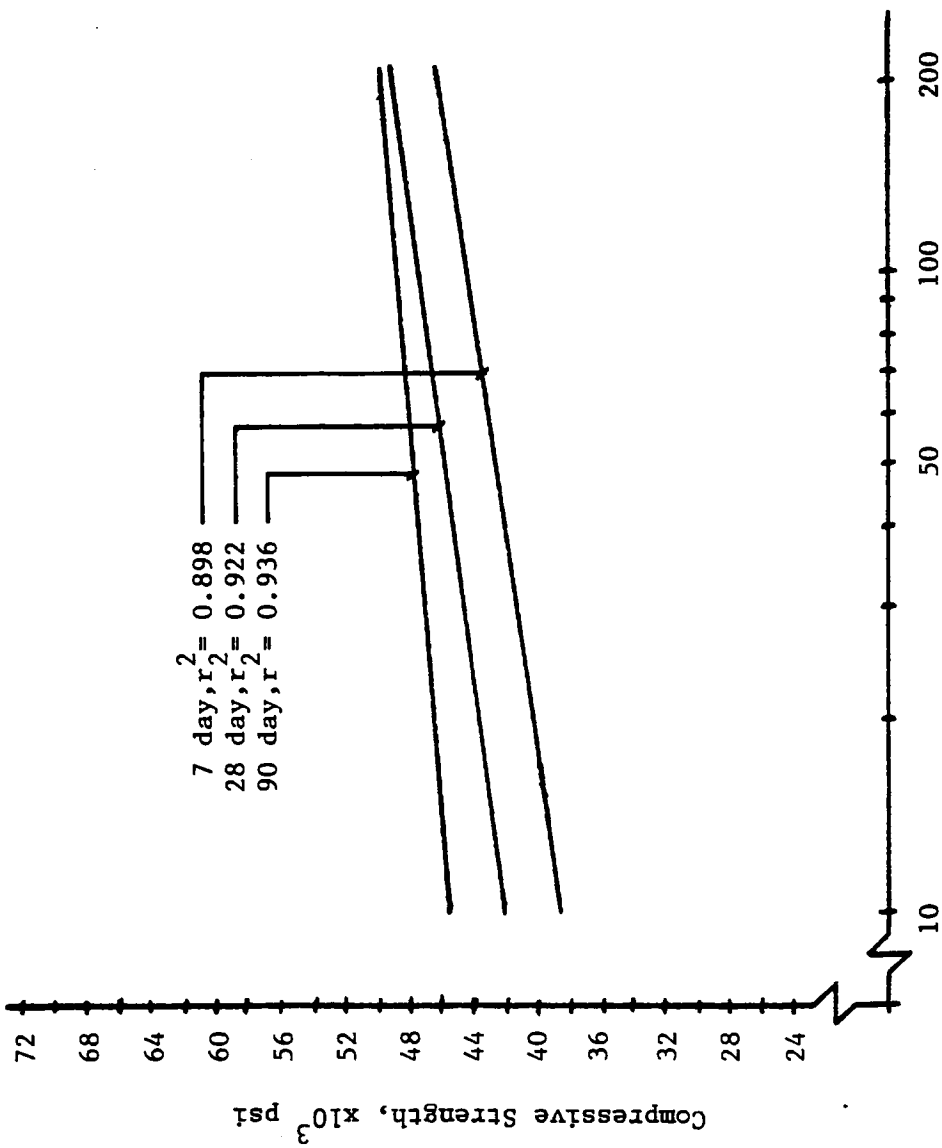

Fig. 21 - Compressive strength vs. production pressure relationship at seven, twenty-eight, and ninety days of age for compacts containing ten percent fly ash (Note: 1 psi = 0.006895 MPa)

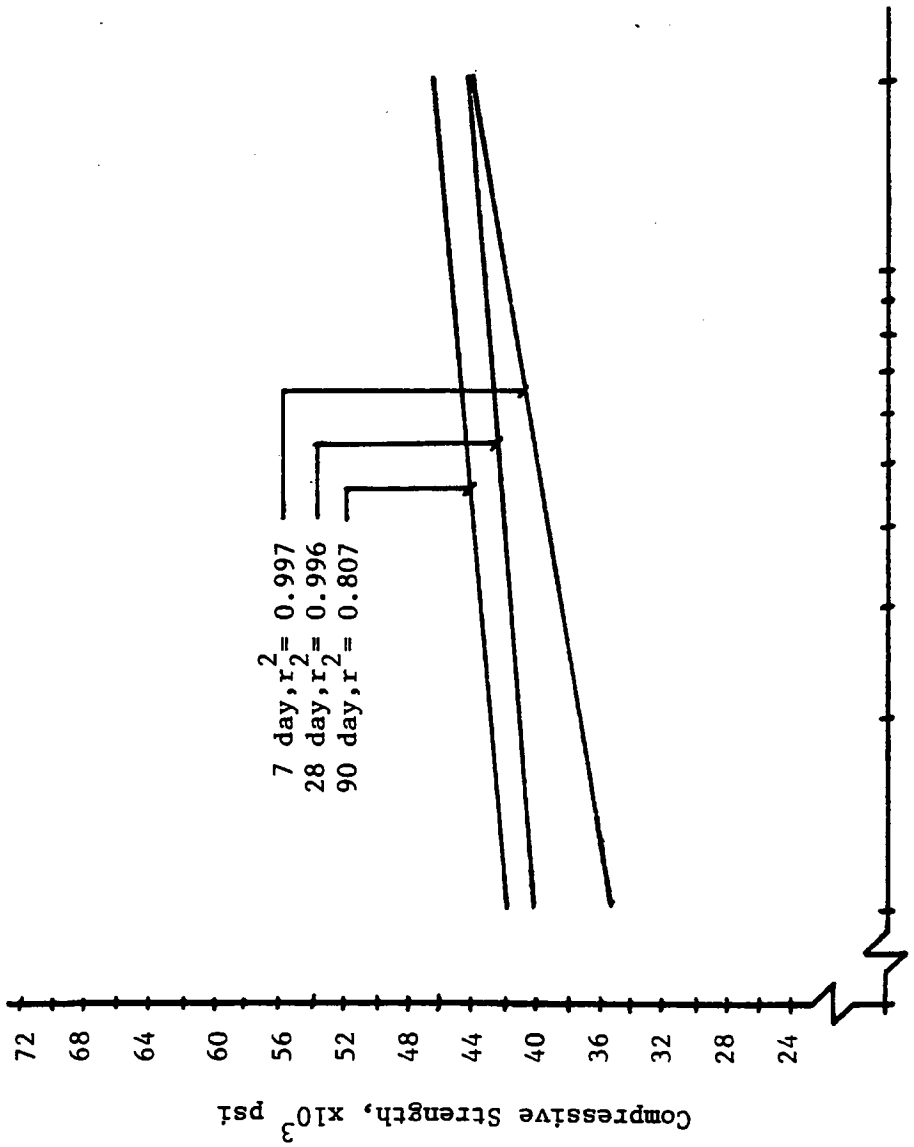

Fig. 22- Compressive strength vs. production pressure relationship at seven, twenty-eight, and ninety days of age for compacts containing twenty percent fly ash
(Note: 1 psi = 0.006895 MPa)

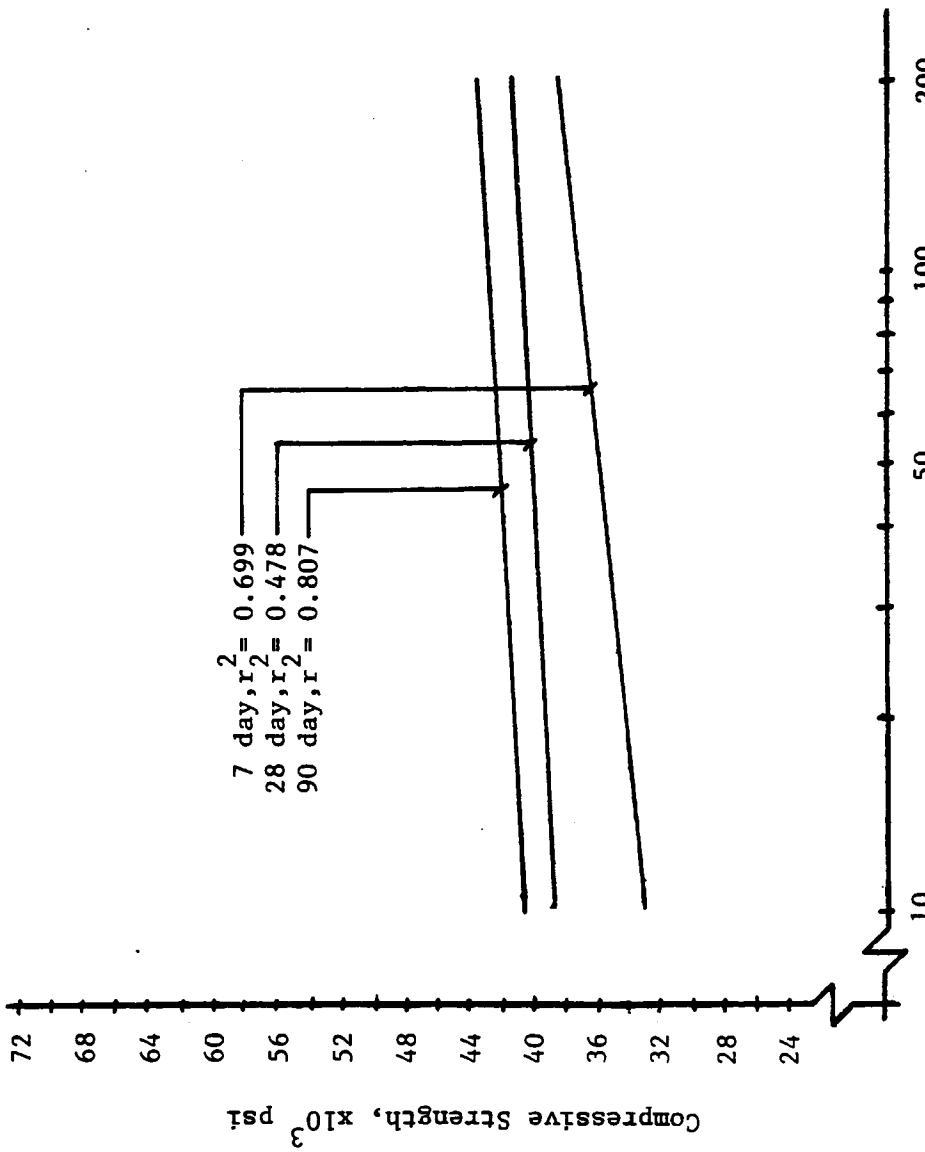

Fig. 23 - Compressive strength vs. production pressure relationship at seven, twenty-eight, and ninety days of age for compacts containing thirty percent fly ash
(Note: 1 psi = 0.006895 MPa)

Fig. 21, 22, and 23 show the compressive strength to production pressure relationships which resulted for the various percentages of fly ash replacement for cement. The same general trends that existed for the cement-only compacts is again evident for the increasing percentages of fly ash. As curing duration is increased, the compressive strength also increases regardless of the percentage of fly ash. From these figures the increasing strengths at the lower production pressures due to hydration product formation is clearly evident. As the percentage of fly ash is increased, the strength at the lower production pressures more closely approaches that of the compacts prepared at the highest production pressures and cured for the same amount of time. Evidently as the percentage of fly ash is increased, the relationship of a higher pressure compaction producing a higher strength material diminishes. This is particularly evident for the longer curing durations as the amount of fly ash is increased.

Fig. 24, 25, 26, and 27 show another interesting aspect of the high pressure compaction effects and the hydration process of the cement material. As discussed, greater amounts of hydration product were believed formed at the higher initial porosity conditions and lower production pressures. These figures show that the increased amount of hydration product formation also has a positive effect on relative increase in the compressive strength of the prepared compacts. The influence of curing duration on increasing strength is also obvious from these figures. It is important to point out, however, that in all cases, the compressive strengths of the compacts

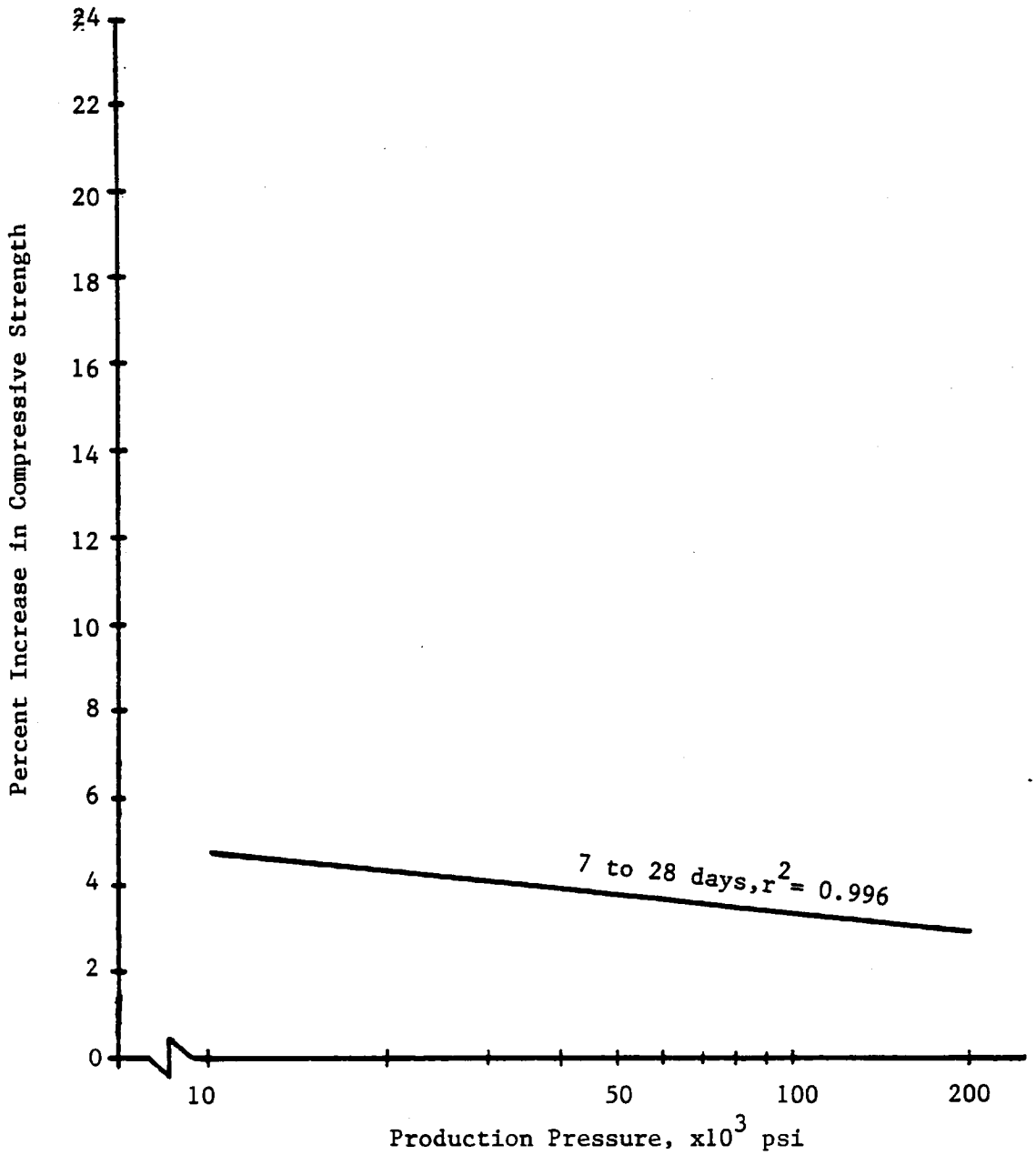

Fig.24 - Percent increase in compressive strength from seven to twenty-eight days vs. production pressure relationship for 100 percent portland cement compacts
(Note: 1 psi = 0.006895 MPa)

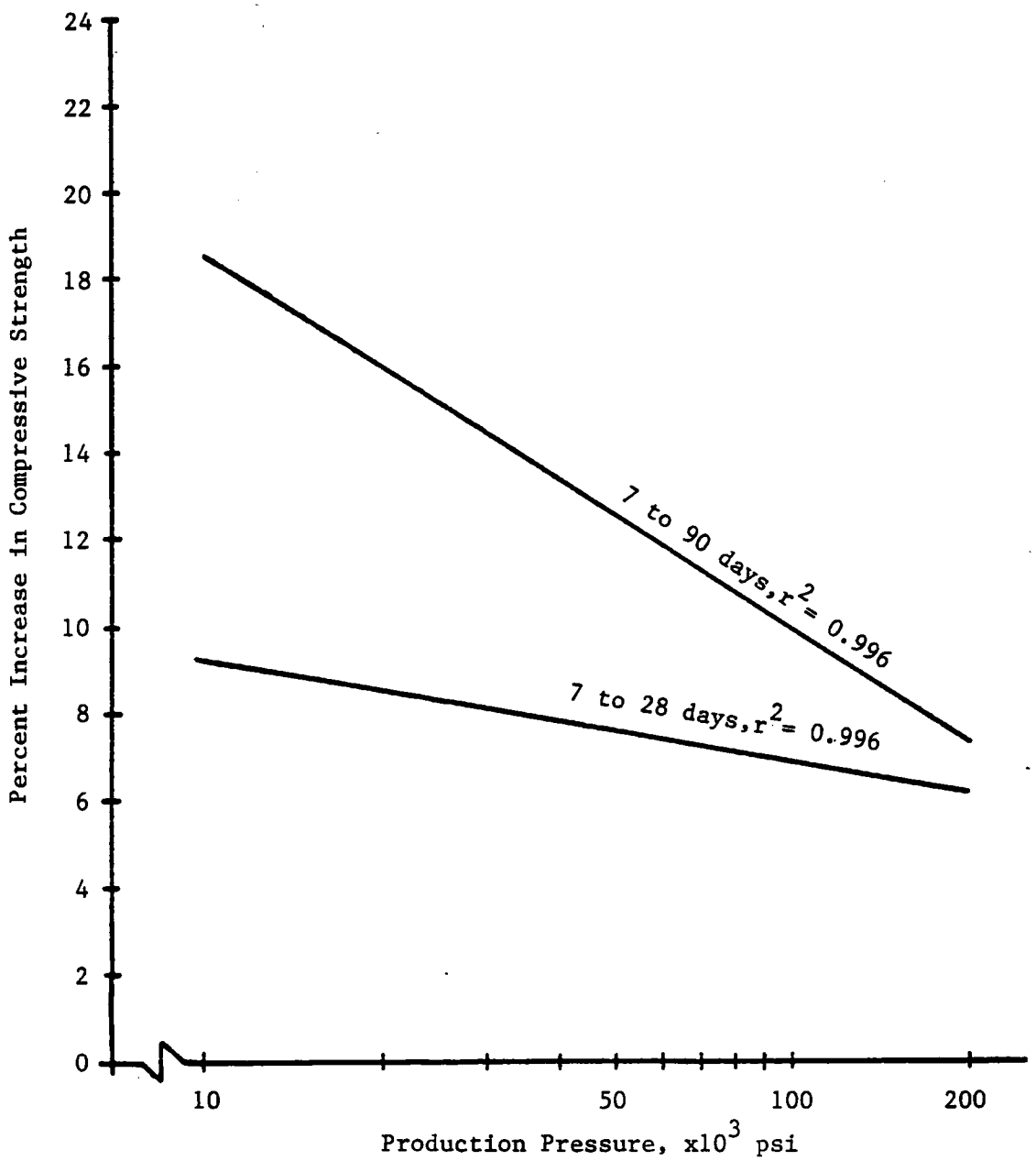

Fig. 25 - Percent increase in compressive strength from seven to twenty-eight and seven to ninety days vs. production pressure relationships for compacts containing ten percent fly ash (Note: 1 psi = 0.006895 MPa)

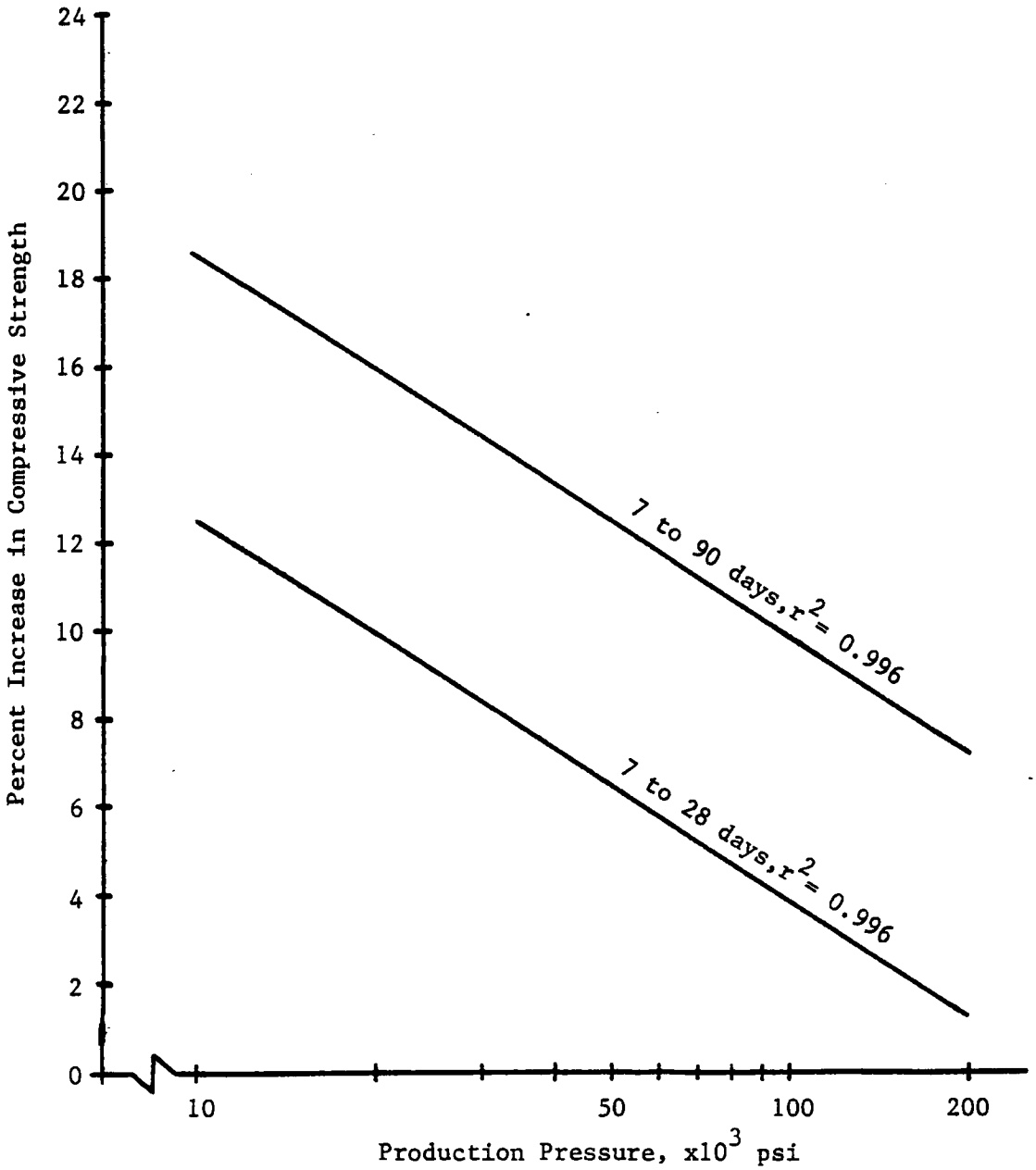

Fig. 26- Percent increase in compressive strength from seven to twenty-eight and seven to ninety days vs. production pressure relationhsips for compacts containing twenty percent fly ash (Note: 1 psi = 0.006895 MPa)

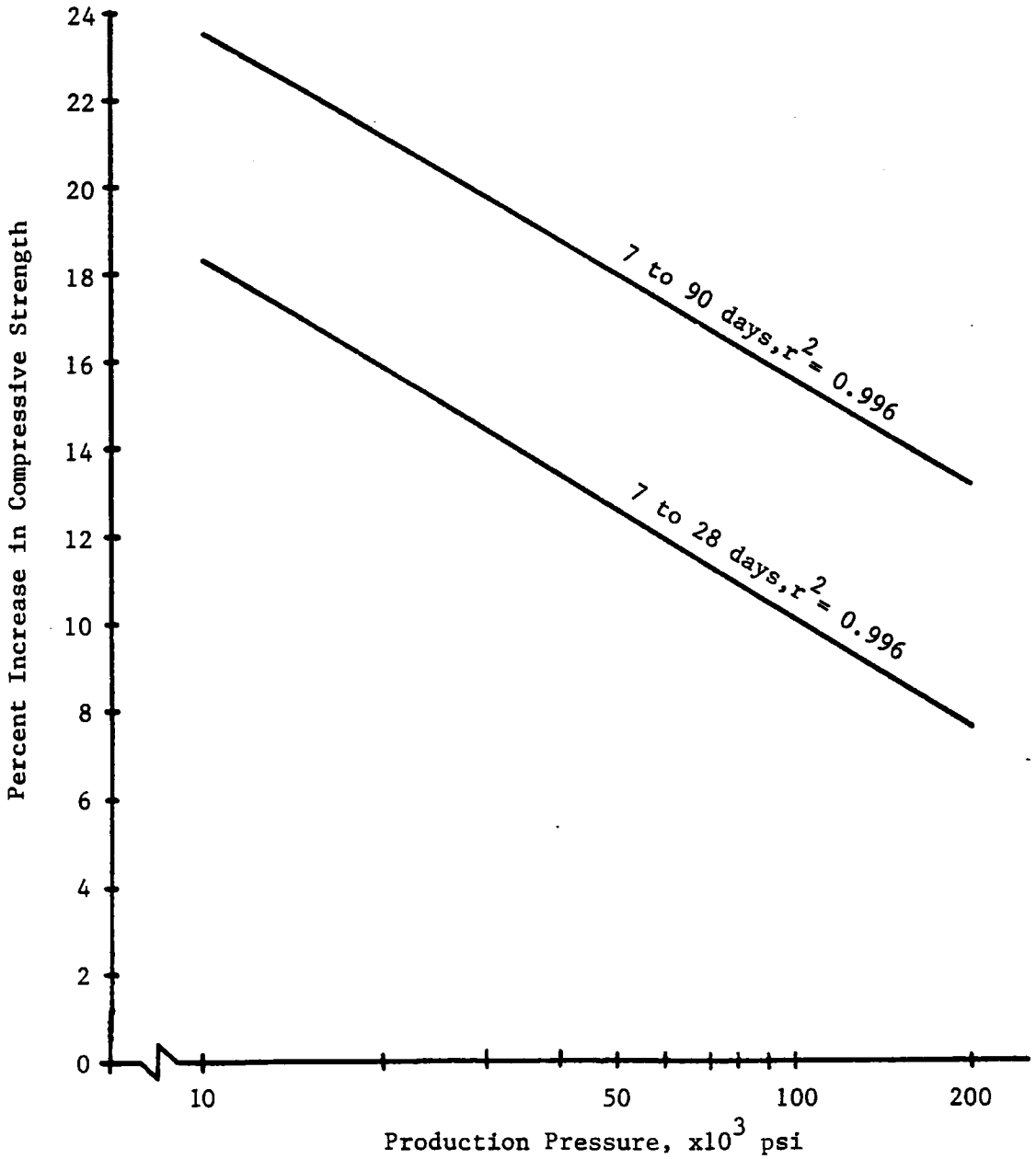

Fig. 27 - Percent increase in compressive strength from seven to twenty-eight and seven to ninety days vs. production pressure relationships for compacts containing thirty percent fly ash (Note: 1 psi = 0.006895 MPa)

prepared at the higher compaction pressures was greater than the compacts prepared at lower production pressures even without the greater formation of hydration products.

Generally, Fig. 24, 25, 26, and 27 show that from either 7 to 28 days, or 7 to 90 days, the compacts prepared at the lowest production pressures experienced the greatest percent increase in compressive strength. This can be misinterpretated because the compressive strengths of the compacts prepared at higher production pressures are already extremely high due to the mechanical reduction of porosity. Thus the resulting percent change in strength is then low. On a relative basis however, these figures show the tremendous influence which the increased formation of hydration products has on the compressive strength.

Although the previous discussion concerned the relationship between compressive strength and production pressure, the figures essentially described the relationship between strength and initial porosity.

The next step is to examine the relationship between the final porosity in the compacts at the time of failure and the compressive strength which was achieved. Unlike the previous figures which were presented in such a way as to present the influence of curing duration on the compressive strength, the following figures showing the relationship between final porosity and compressive strength are presented such that the influence of the fly ash on the compressive strength will be clearly evident.

Fig. 28 shows the influence of final porosity on the 7 day

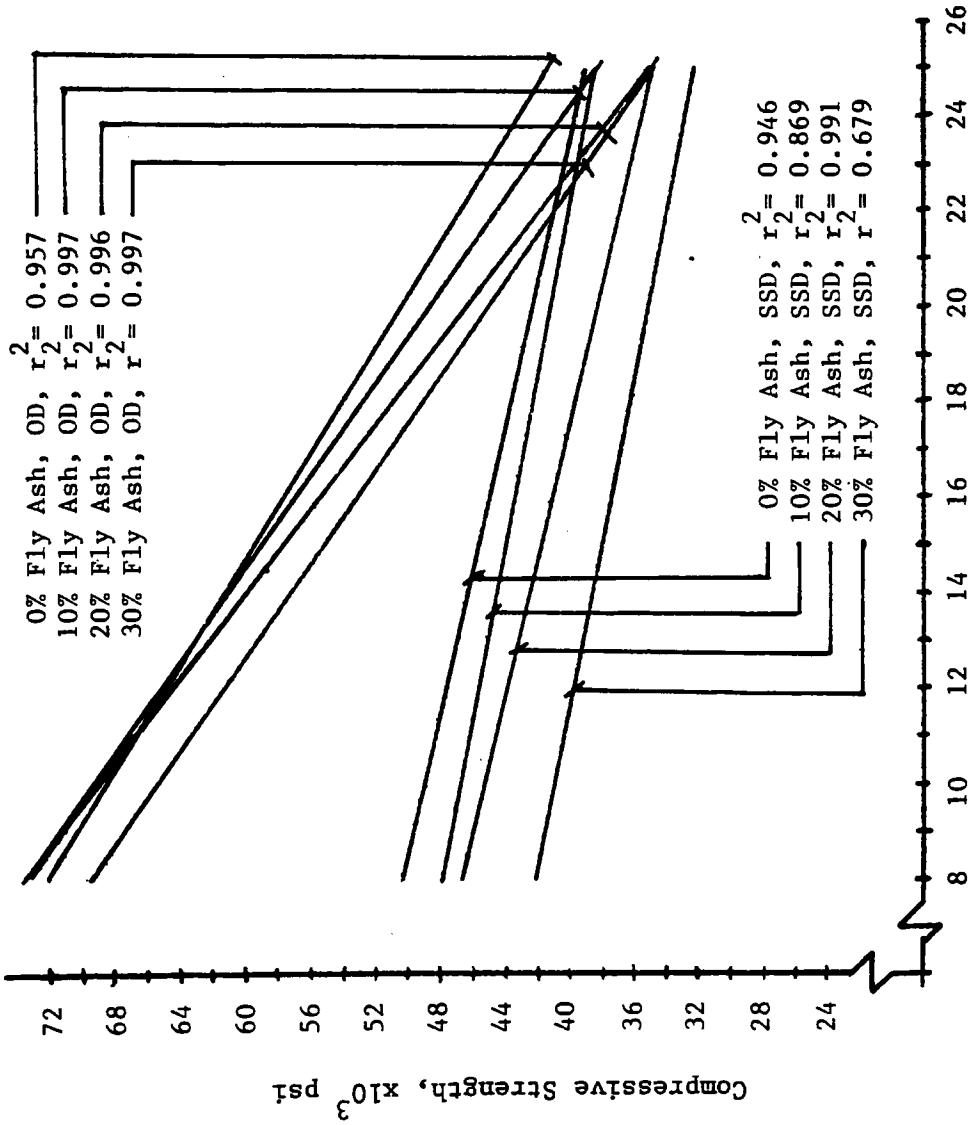

Fig. 28 - SSD and OD compressive strength vs. final porosity relationships at seven days of age for compacts containing zero, ten, twenty and thirty percent fly ash (Note: 1 psi = 0.006895 MPa)

compressive strength of compacts containing various percentages of fly ash. Quite noticible is that increasing the percentage of fly ash in the compacts resulted in decreasing compressive strengths. This is a reasonable result at this early age due to the fact that the pozzolanic reaction is slow in developing and because less cement is present. It is interesting to note, by the parallel relationships of the lines, the equality of the strength reductions as the percentage fly ash is increased for all levels of porosity shown.

For reference purposes only, the compressive strengths achieved in the compacts which were oven dried are included on this figure. Note the tremendous apparent increase in strength of the compacts due to the oven drying procedure. Furthermore note the pronounced effects of low levels of porosity on compact strength. The greatest increase in apparent strength occurred at the lower porosities, which could mean that the greatest restrained shrinkage stress developed in the low porosity specimens. In typical concretes, current theory however is such that low porosity conditions provide for less shrinkage of the material and therefore formation of less restrained shrinkage stress. Because of this, the large apparent increase in strength can not be attributed wholly to the formation of restrained shrinkage stress. Another possible reason for this strength - porosity relationship in the oven dried compacts is the formation of additional, very influential strength-producing hydration product by accelerated curing during the oven drying process. This would explain the larger apparent increase in strength at the lower porosity

conditions than at the higher porosity conditions.

Fig. 29 shows the influence of final porosity on the compressive strength at 28 days of curing. Again as at the 7 day age, increasing amounts of fly ash reduce the compressive strength at all porosity levels. Observing the saturated surface dry data, there does appear to be a more significant increase in strength for the 0 and 10 percent amounts of fly ash than for the 20 and 30 percent amounts of fly ash material. This figure suggests that the replacement of cement with fly ash material in the high pressure compaction process up to about 10 percent by weight has very little effect on 28 day strength. With replacements over 20 percent, however, the reduction in strength becomes substantial at 28 days. Observing the oven dried data, again presented for reference purposes, this trend in strength to percent fly ash is supported as the compacts containing 30 percent fly ash exhibited much lower apparent strengths than the compacts containing lesser amounts of fly ash.

Fig. 30 shows the relationship which existed at 90 days of curing between the final porosity and compressive strength. Unfortunately compressive strength data for cement only was unavailable at 90 days. However the same general trends that existed for the 7 and 28 days of age appear to exist at 90 days of curing as the increasing percentages of fly ash resulted in decreasing compressive strengths. For the higher porosity conditions, a similar result to that found at 28 days is observed for amounts of fly ash greater than 20 percent. At the lower porosity conditions this point is not as obvious. Again observing the oven dried apparent strengths, particularly at the lower

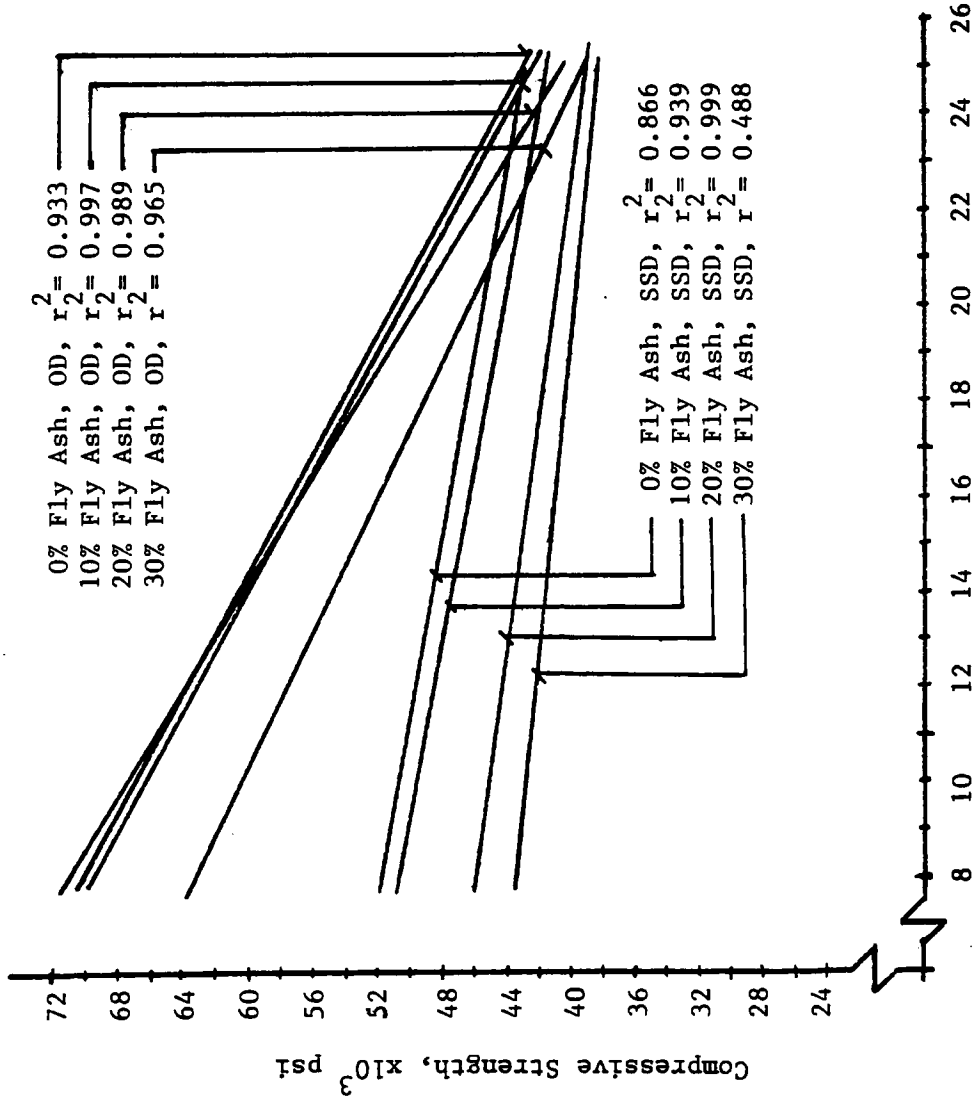

Fig. 29 - SSD and OD compressive strength vs. final porosity relationships at twenty-eight days of age for compacts containing zero, ten, twenty, and thirty percent fly ash (Note: 1 psi = 0.006895 MPa)

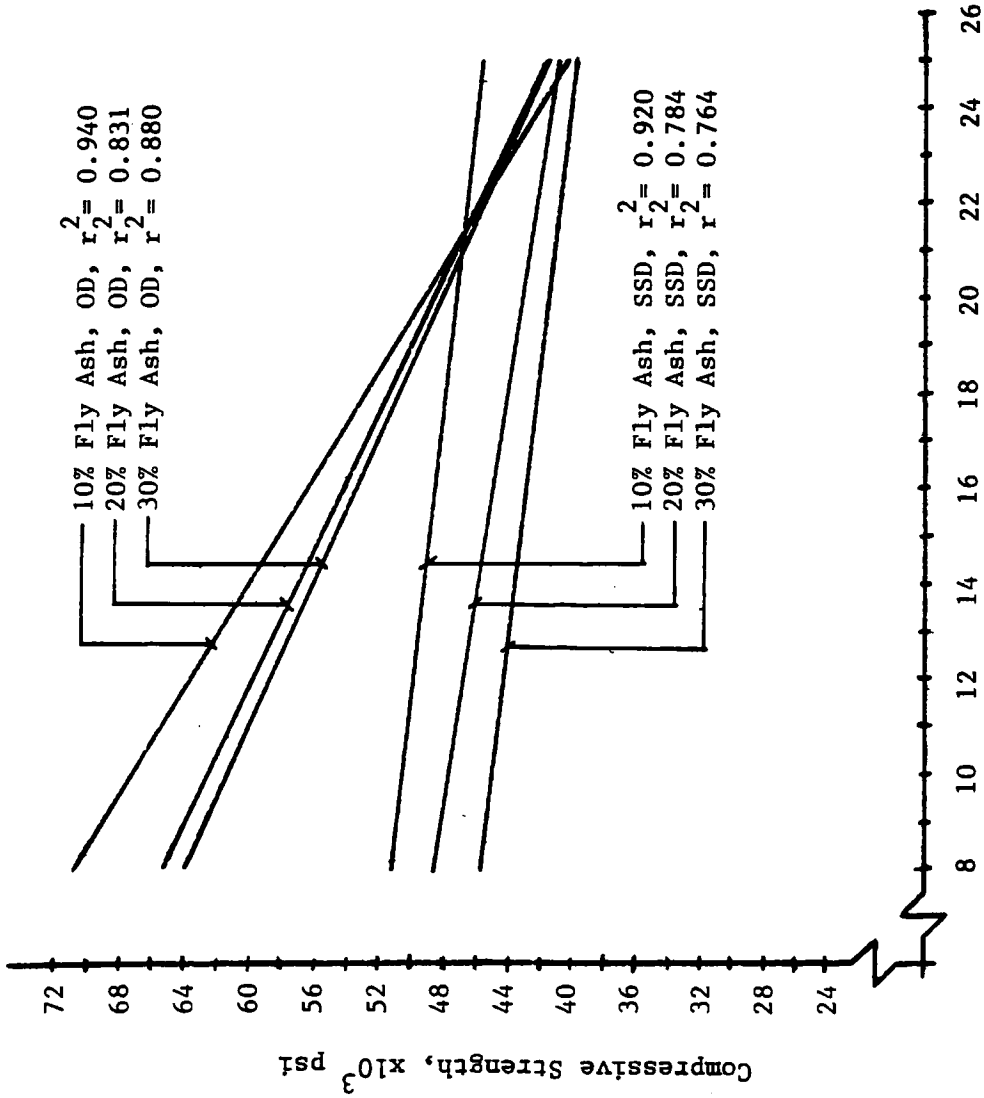

Fig. 30 - SSD and OD compressive strength vs. final porosity relationships at ninety days of age for compacts containing ten, twenty, and thirty percent fly ash (Note: 1 psi = 0.006895 MPa)

porosities, the delineation between 10 and 20 percent amounts of fly ash producing very different strengths is visible.

At the initial outset of the laboratory investigation, the influence of fly ash on the strength of the prepared compacts was of primary importance. As shown in various discussions to this point, the fly ash had an adverse effect on the strength of the compacts at all percentages of replacement. Fig. 31 shows the marked effects of fly ash on the reduction of strength in the prepared compacts at all ages investigated. This is also a normalized relationship in that the compressive strengths used for determination of the relationships included all production pressures. The control for this figure was the strength determined by the linear regression relationship for compressive strength versus production pressure for zero percent fly ash, 7 day curing.

Each line shown on the figure is the result of a linear regression analysis of the percent change in compressive strength from the control strength at a particular production pressure. Generally the figure shows that for all production pressures or initial porosity conditions, the compressive strength increased from 7 to 90 days approximately 12 percent of the 7 day strength regardless of the percentage of fly ash in the compact. The figure also shows that for each ten percent of fly ash replaced in the compacts, 6 to 7 percent of the compressive strength was lost at all curing durations regardless of production pressure.

To further make a point concerning the influence of fly ash, it is

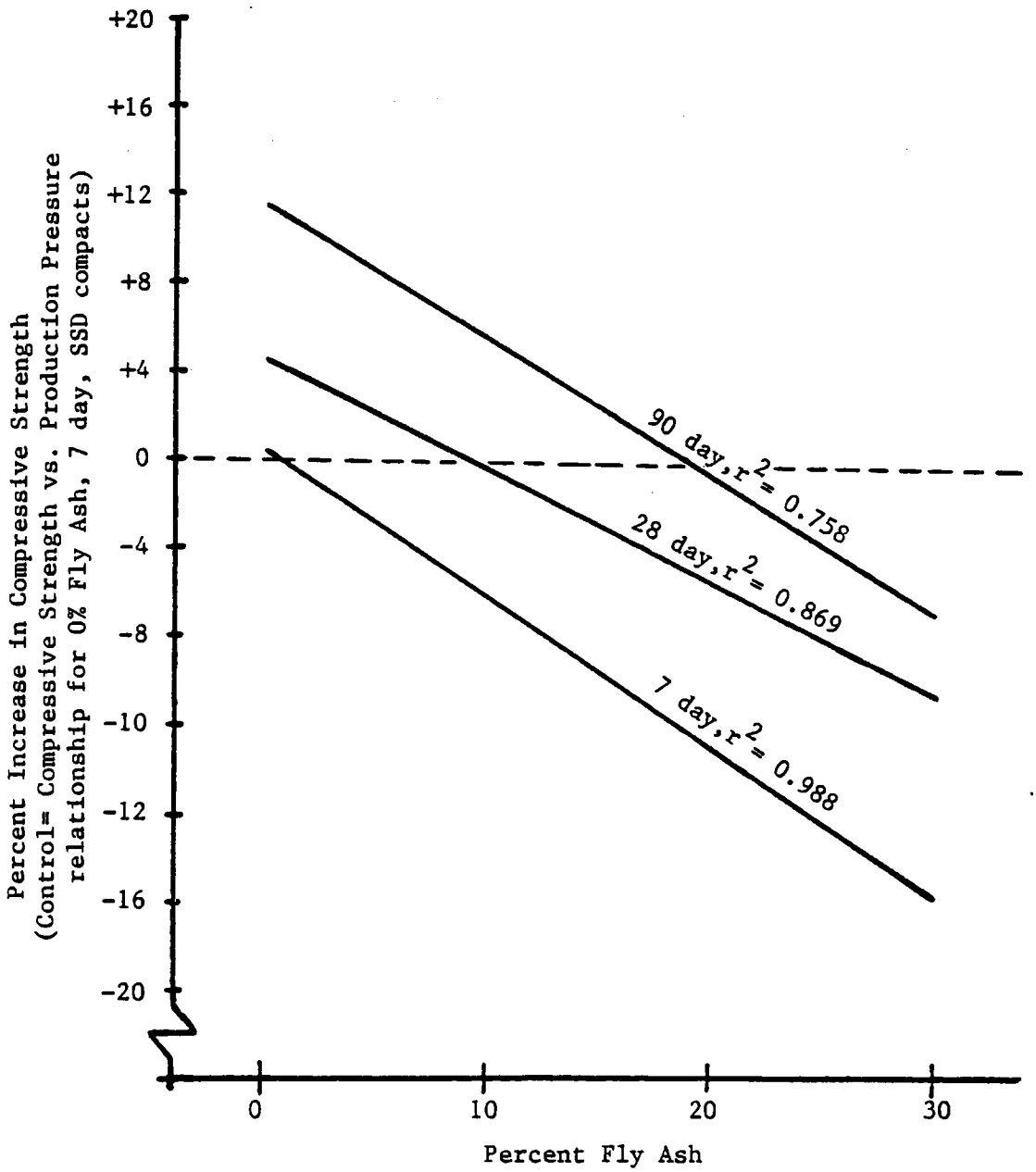

Fig. 31 - Percent increase in compressive strength vs. percentage fly ash replacement relationships at seven, twenty-eight, and ninety days of age

appropriate to enter the figure at the control point of zero percent increase in compressive strength and compare the strengths of the various percentages of fly ash at the various durations. From this exercise, at approximately 10 percent fly ash cured for 28 days, the strength is the same for zero percent fly ash cured for 7 days. Likewise for the 20 percent fly ash value, 90 days curing is required to achieve the same compressive strength as zero percent fly ash cured for 7 days. For the 30 percent fly ash value, the curing periods up to 90 days were insufficient to produce strengths equal to 7 day strengths of cement only.

In summary it appears in terms of compressive strength, that, no optimum percentage of fly ash existed for up to 90 days of moist curing. At least no particular percentage of fly ash resulted in significant increases in strength over that of the cement only compacts. At all durations up to 90 days, increasing percentages of fly ash produced incrementally lower compressive strengths. It appears, although not conclusively by any means, that fly ash replacement up to 10 percent had a less significant effect on reducing strength than replacement percentages of greater than 20 percent. Perhaps the 90 day curing duration utilized was an insufficient amount of time to allow for the pozzolanic reaction to fully become established or, as discussed earlier, the high pressure compaction process had a negative effect on the pozzolanic reaction.

Because of the excellent linear relationships which existed for all of the various parameters involved in the high pressure compaction of the portland cement and fly ash, multiple linear regression

analyses were performed. The results of these analyses were excellent and provided useful mathematical models of the compressive strength which could be obtained with the combination of the various parameters.

The resulting equation determined by this analysis by combining all of the various parameters is:

$$f'c = -303.8\ FA + 4401.7\ \mathrm{Log}_{10}\ Age + 4441.5\ \mathrm{Log}_{10}\ PP - 497.1\ P_i - 2666.4\ P_f - 66072.5\ Den_i - 26286.3\ Den_f + 291693.0 \qquad (6)$$

where f'c is the predicted compressive strength based on the parameter values supplied,

FA is the percentage of fly ash in the compact in percent,

Log_{10} Age is the base 10 logarithm of the curing age in days,

Log_{10} PP is the base 10 logarithm of the production pressure in psi,

P_i is the initial porosity of the compact in percent,

P_f is the final porosity of the compact in percent,

Den_i is the initial density of the compact in grams per cubic centimeter,

Den_f is the final density of the compact after removal of free water by oven drying at 220 F (105 C) for 48 hours, in units of grams per cubic centimeter.

The multiple R or the multiple coefficient of correlation for this equation is 0.950. It is important to point out that this equation was developed from all the SSD data obtained from the various series.

Concerning this equation, there exist two important points which need to be made. The first is that logarithmic values of the curing duration and production pressure must be used with the other data collected to obtain a high degree of correlation. The second point is the fact that this equation was developed from a very definite range and combination of values as contained in Appendix E. For this equation to result in realistic strength predictions, the boundaries and relationships upon which it is based must be followed.

Chief among these relationships is the influence of production pressure on the porosity and density conditions as shown previously in Fig. 12, 13, 14, 15, 16, and 17. The multiple regression equation discussed here is based on the relationships shown in these figures. In the most simplistic terms, the equation is based on the fact that higher production pressures will produce corresponding lower porosity conditions and resulting higher densities. Concerning typical values, the above mentioned figures should be consulted. For a production pressure of approximately 185,000 psi (1,275 MPa), the initial porosity used must be in the 20 to 21 percent range with an initial density in the range of 2.4 to 2.5 g/cm^3. Conversely, for a production pressure of approximately 14,000 to 15,000 psi, a corresponding initial porosity of 32 to 33 percent and initial density of 2.0 to 2.1 g/cm^3 must be used for correct use of the equation.

Realistic values for the final porosity and final density, depending on the curing duration and percentage of fly ash must also be used if reliable strengths predictions for prepared compacts are to be made.

Whereas the previous equation defines the entire system and results of the data collected from the laboratory investigation, perhaps a more useful predictive equation of the strength of compacts prepared with the die would only utilize the various initial parameters involved.

Using this thought, the following equation was developed by multiple linear regression:

$$f'c = -150.6\ FA + 6391.1\ \text{Log}_{10}\ \text{Age} - 8601.9\ \text{Log}_{10}\ PP + 39211.9\ \text{Den}_i - 10971.9 \quad (7)$$

The multiple R or coefficient of multiple correlation for this particular equation is 0.932. Again, as with the previous equation, this equation was developed from data on all the SSD compacts and series tested.

Again, the relationships discussed for the previous equation also apply to this particular one. The use of a realistic relationship between a higher production pressure producing a greater initial density is extremely important for a correct compressive strength prediction of prepared compacts with this equation. Fig. 14, 15, 16, and 17 show the typical relationships resulting between production pressure and initial density achieved depending on the

percentage of fly ash. It is interesting to note that initial porosity was not included in this prediction equation. This is due the excellent inverse relationship which exists between porosity and the production pressure and the inclusion of both would essentially be a duplication of initial parameters. Parameters chosen for use in this equation have values which are also readily available once a compact has been prepared and make this equation quite useful as a compressive strength prediction equation for the miniature compacts.

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

The following presents a summary of the conclusions made from an analysis of the literature surveyed and the laboratory investigation performed.

1. High pressure compaction is an excellent means of producing a low porosity condition and ultimately a greater strength in a cementitious material.

2. The level of porosity present in prepared portland cement compacts, both initially and at the completion of curing, has a linear relationship with the ultimate compressive strength for the 20 to 32 percent initial porosity and resulting 8 to 20 percent final porosity range.

3. From the laboratory investigation, in all instances, the higher the initial porosity condition, the greater was the amount of porosity present upon completion of curing. For compacts with a greater initial porosity, a greater amount of hydration product formed which resulted in a much greater percentage strength increase in the compact.

4. In all instances the greater reduction of porosity due to high pressure compaction resulted in a greater strength than a lesser amount of compaction with its resulting higher porosity and subsequent increased hydration product formation.

5. Over time, the strength of the compacts initially prepared

at higher initial porosities appear to approach the strengths of the compacts prepared at initially lower porosity levels.

6. The high pressure consolidation of portland cement material and a Class "C" fly ash material can be accomplished with excellent results. Compatibility of the materials, however, is one area which must be considered in the preparation of compacts containing fly ash. It was found that the use of a fly ash material passing a No. 100 sieve produced a more stable compact at all percentages of fly ash replacement for cement than the use of the coarser raw fly ash material furnished.

7. The existence of an optimum percentage of fly ash replacement of the cement in the miniature compacts could not be determined. The influence of the fly ash on the compressive strength of the prepared compacts was negative in that the increasing replacement of the cement with fly ash resulted in decreasing compressive strengths at all ages of curing. On a percentage basis, approximately 5 to 6 percent loss in strength was experienced at all durations with each ten percent replacement of cement with fly ash.

8. The expected increased strength due to the pozzolanic reaction was not clearly evident at all percentages of fly ash replacement. Evidently the pozzolanic reaction between the fly ash and the calcium hydroxide of the cement when combined in a low porosity condition require longer amounts of time for formation or the high pressure compaction process hindered the formation of needed calcium hydroxide and the expected pozzolanic reaction was in turn

affected.

9. End cap failure of the compacts upon release of the production pressure was a recurring problem during the preparation of the compacts for this project. Although the use of sieved fly ash material eliminated the cause of some of the problem, end cap failures of the freshly prepared compacts were still quite numerous as the percentage of fly ash in the compacts increased. For cement only compacts this problem was virtually non-existent.

10. Oven drying of the prepared compacts to determine the amount of free water present as a measure of the final porosity produced significant apparent strength increases in the compacts tested, probably due in large part to the creation of restrained shrinkage stresses.

11. From the data collected concerning initial and final porosity, the various densities determined, the production pressure, percentage of fly ash replacement and the curing duration, a multiple regression analysis was performed to determine a resulting compressive strength equation for the small cubicle compacts based on the laboratory procedures used. This equation is:

$$f'c = -303.8\ FA + 4401.7\ \text{Log}_{10}\ Age + 4441.5\ \text{Log}_{10}\ PP - 497.1\ P_i - 2666.4\ P_f - 66072.5\ Den_i - 26286.3\ Den_f + 291693.0 \qquad (6)$$

The multiple coefficient of correlation for this equation is 0.950.

12. Whereas the previous equation defines the entire system and

results of data collected from the laboratory investigation, a more useful predictive equation of the strength of the compacts prepared with the die was developed by multiple linear regression which utilizes only the various initial parameters such as the intended age of curing, the percentage of fly ash in the compact, the production pressure used to produce the compact and the resulting initial density of the anhydrous compact. The resulting predictive equation is:

$$f'c = -150.6\ FA + 6391.1\ \text{Log}_{10}\ \text{Age} - 8601.9\ \text{Log}_{10}\ PP + 39211.9\ \text{Den}_i - 10971.9 \quad (7)$$

The multiple coefficient of correlation for this equation is 0.932.

13. These compressive strength prediction equations are only valid for a certain range of values. Since the equations are essentially a mathematical model of the entire system utilized in this project, specific relationships between production pressure and the resulting porosity and density conditions must be taken into account for the attaining of proper values with these equations.

Recommendations

The following recommendations pertain to the laboratory performance portion of the work performed for this project and are intended to aid in the more effective and accurate determination of data for future work involving the use of the die used for this project.

1. Whereas for this project the powder material was mixed in numerous smaller portions when needed, it is suggested that at the initial outset of the laboratory investigation an adequate stock supply of material containing the appropriate amount of component materials be mechanically mixed and properly stored for future use. This would provide for better quality laboratory results in two ways. One, the potential for error in the combination of the proper amounts of powder material is eliminated, if done properly the first time, and an increased consistency in the powder material used for the individual series is achieved. Secondly, by preparing stock quantities of powder material for the various percentages of fly ash replacement, specific gravity determinations can be made for the combined powder material with each percentage of fly ash. In this way a more realistic value of the true specific gravity of the mixed powder materials could be used for porosity determinations.

2. Careful consideration should be given to the order in which the compacts are prepared. For this particular study, all compacts for a particular duration were prepared before the compacts of another duration were prepared. Because of the data variability which exists in the preparation of the compacts in this way, the determination of successive changes in porosities and densities for the various durations was not possible. Excellent results, however, were obtained at each duration when the compacts cured for that duration where compared for porosity and density change relationships. The point here is that due to data variability, the actual intent of the laboratory investigation should be considered when determining the

order in which to prepare the compacts.

3. Even though excellent relationships were determined concerning compressive strength and the various other parameters, it is the opinion of the author that the strengths results in terms of consistency and uniformity could be improved with the use of a special device designed specifically for use in the failure of the miniature cubicle compacts.

4. From the results obtained, the use of a precuring procedure whereby the freshly prepared compacts are exposed to a fine mist for initial periods of hydration and compact stabilization before complete immersion would be beneficial in reducing the quantity of compacts which fail prematurely in the curing solution.

Future Research

The following discussion concerning other possible research topics was developed during the course of performing this study. Each of the items discussed has relative importance to the preparation of high strength materials through high pressure compaction.

1. Since the initial intent of this study was to determine the effects of fly ash on producing a higher strength material due to the pozzolanic reaction, the logical area for future work is to increase the period of time which the compacts are moist cured in an effort to allow the expected pozzolanic reaction to fully occur.

2. Also concerning this point, additional work as to the effects of high pressure compaction on the pozzolanic reaction and the

amount of calcium hydroxide available for the reaction might provide valuable insight into the feasibility of even utilizing fly ash in a high pressure compaction system.

3. As shown by this study, particle compatibility in the compaction process is important. Considerable work needs to be done as to the proper combination of a range of particles sizes to produce a higher strength material through high pressure compaction. Determination of the degree of particle crushing which occurs at the high production pressures is also critical to this determination of the optimum particle gradation involved in the high pressure compaction process.

4. Considerable work also remains pertaining to the amount and type of hydration products which are formed in compacts having low porosity conditions.

5. If the ultimate goal is the production of the greatest possible strength achievable in a cemetitious material, considerably more effort could be directed at the effects of oven drying and the formation of restrained shrinkage stresses on the production of a higher strength material. Purely visionary and hypothetical at this point, the author finds it intriguing that perhaps a system producing small blocks or bricks could be designed whereby a cementitious material with an extremely low porosity conditon is produced through high pressure compaction. Upon completion of curing, the small blocks are then oven dried to produce the restrained shrinkage stress and a sealant material applied to keep the block in the moisture free condition and thereby possess ultra-high strength indefinately.

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APPENDIX A

CEMENT AND FLY ASH DATA

Cement Composition and Properties

Midlothian TXI Type III Cement

Physical Test Results as reported by TXI

NC	26.5
% H_2O Cubes	48.5
% Flow	112
% Air	9.0
% Passing #325 Sieve	98.6
Blaine	535
Wagner	255

Gilmore Setting Time Initial/Final	2:35/4:15
Vicat Setting Time Initial/Final	0:50/3:20
% Fed. False Set	7.1
Autoclave Expansion	

D.O.P.	
1	50
2	50
3	50
4	50
5	50

Compressive Strengths (2"x2" cubes)	
1 day	3592 psi
3 day	5425 psi
7 day	6242 psi
28 day	7575 psi

Specific Gravity	3.13

Cement Composition and Properties continued,

Chemical Analysis / X-Ray Analysis

SiO_2	20.11
Al_2O_3	4.38
Fe_2O_3	3.52
CaO	64.66
MgO	0.78
SO_3	3.34
P_2O_5	0.23
TiO_2	0.22
Cr_2O_3	0.00
Mn_2O_3	0.30
Na_2O	0.29
K_2O	0.38
Total	98.20

C_3S	63.40
C_2S	9.80
C_3A	6.80
C_4AF	10.70

C_3A calculations include Al_2O_3, P_2O_5, and TiO_2

Fly Ash Composition and Properties

Gifford-Hill Class "C" Fly Ash

Physical properties for the unsieved fly ash material as reported by the Gifford-Hill Company,

	Sample	Specification
Fineness, +325 Sieve, % Retained:	18.3	34.0 max.
PAI (28 days), % Control	101.4	75.0 min.
Water Requirement, % Control	90	105.0 max.
Autoclave Soundness, %	0.218	0.8 max.

Physical properties for the unsieved fly ash material as reported by the Gifford-Hill Company continued,

		Sample	Specification
Specific Gravity:	unsieved	2.71	....
	sieved*	2.75	....

Chemical properties for the unsieved fly ash material as reported by the Gifford-Hill Company,

	Sample	Specification
Silicon Dioxide	32.03	
Aluminum Oxide	17.10	
Ferric Oxide	7.25	
Total	56.38	50.0 min.
Calcium Oxide	29.88	
Magnesium Oxide	6.34	
Sulfur Trioxide	3.61	5.0 max.
Sodium Oxide	2.08	
Potassium Oxide	0.26	
Loss on Ignition	0.88	6.0 max.
Moisture Content	0.07	3.0 max.
Total	99.50	
Sodium Oxide Equivalent	2.25	

Note: (*) fly ash passing No. 100 sieve as used for the laboratory investigation.

APPENDIX B

LABORATORY PROCEDURES FOR THE HIGH PRESSURE PREPARATION OF CEMENT AND FLY ASH COMPACTS

Introduction

The procedures detailed in this appendix are a step-by-step approach to the preparation, curing and testing of small cubicle compacts composed of portland cement and fly ash materials by high pressure consolidation. As discussed in the report, the die equipment used for this project was designed by Dr. Matti Relis. The nature of the testing procedures used are simple. The steps followed are quite repetitive however and become a monotonous process over time. Good results are dependent on avoiding the pitfalls associated with a monotonous, repetitive process. A consistency in performing the various procedures is also very important for good results and was the main objective upon initial familiarization to the testing process.

Preparation of the Powder Material

As discussed in Chapter III, the first step of the testing process was preparation of the material to be compacted. For compacts consisting of cement only, this step of the process was not necessary.

Once the weight determinations for the proper proportions had been made, the cement and fly ash material were each weighed out in a

seperate container and then combined in a larger container for mixing. From this larger mix of the powder material, the powder needed for each inidividual compact was later taken. Generally 50 to 100 grams of combined powder was mixed at one time and was dependent upon the quantity of compacts to be prepared at that time. To provide for more meaningful data, enough powder was mixed at this point to provide a series of compacts for both the wet surface dry and oven dried test conditions for a given combination of parameters. The percentage of fly ash in the mix also influenced the amount of powder which was initially mixed because of the higher percentage of premature compact failures during the compaction process with increasing amounts of fly ash.

Mixing was performed by the stirring of the powder material with a glass stirring rod until no visible segregation of the powder material existed. This stirring action was followed by the placement of the combined powders in a closed container, allowing for additional mixing by vigorous shaking and tumbling of the powders. Upon numerous repetitions of the procedure, the technician develops a feel for when the powder material is well mixed.

There are two other methods of powder preparation which were not used for this work but deserve mention at this point. One method is the mixing of the powders for each individual compact. This procedure would be extremely time consuming and have a greater probability of error and variation among the individual compacts. The other method is a one time mixing of the cement and fly ash material in large quantity. In this way a tremendous amount of time

could be saved, mechanical mixing could be used, and the consistency in the mixed powder improved. In retrospect, this is the recommended procedure.

Once the desired amount of material was mixed, the next step was the weighing out of the amount of powder needed for each of the individual compacts. This was done on a Sartorious Model 2442 scale which could be read to 0.0001 grams. The powder material for each of the individual compacts was placed in a paper cup container until placement in the die. Fig. 32 shows the various items used for this portion of the laboratory procedure.

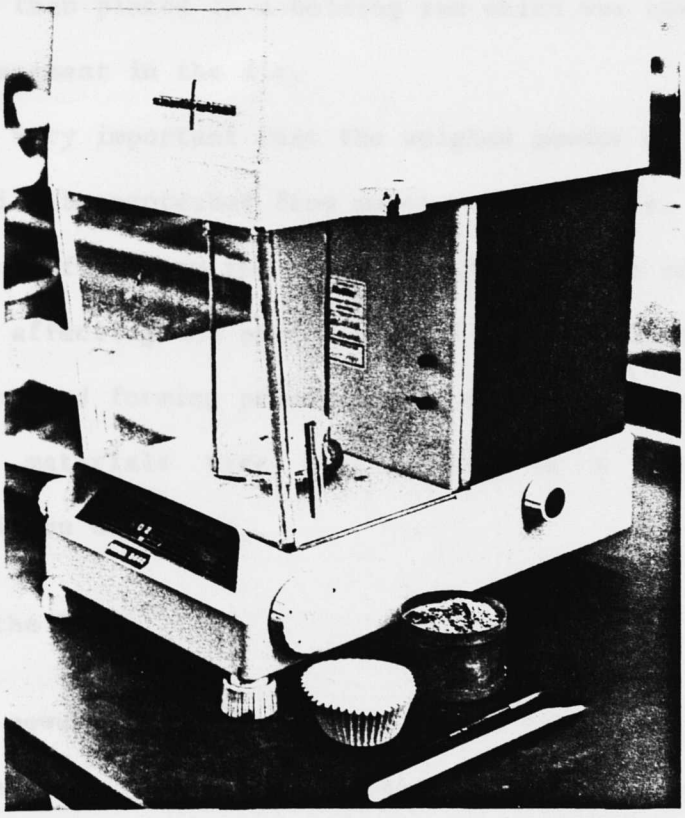

Fig. 32 - Equipment used for weighing the powder material

The procedure followed for the weighing out of the powder material for each of the inidividual compacts was:

1) Checking of the scale to see that it was "zeroed" and in proper working condition.

2) Weighing the empty paper cup container to determine a tare weight.

3) Once the weight of the container was known, the powder material was then added in the proper amount. A small, fine bristled brush was used to add and replace extremely small amounts of powder in order that the exact amount of necessary powder was achieved.

4) Upon weighing, the paper holders containing the powder material were then placed in a holding pan which was covered with tin foil until placement in the die.

It was very important that the weighed powder as well as the supply material be protected from moisture in the air. Moisture can have disastrous effects on the overall quality of the compaction tests performed by affecting the powder weights causing inaccurate density considerations, and forming premature hydration reactions. For these reasons, all materials were kept covered in a minimum moisture exposure condition until use.

Assembly of the Die

Once the powder material for each of the individual compacts was prepared, the next step of the process was the compaction of the material. Fig. 33 shows an exploded, disassembled view of this die. As stated in the report, the compaction of the powder material occurs

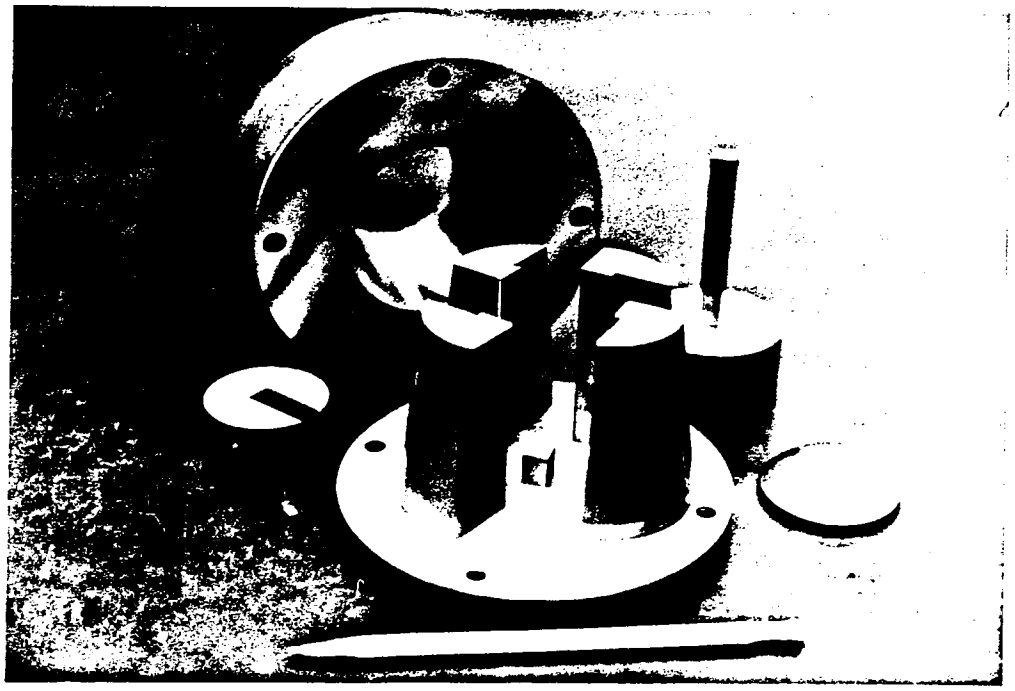

Fig. 33 - Disassembled view of the die

as a compactive force is applied to a ram placed in the die. For this process to occur successfully, the die must be assembled correctly for each and every compact.

Because the particular die used has a segmental design, the tooled fit pieces of the die are held together with the aid of a clamp ring and high tensile strength bolts. Assembly of these segments in the proper order is important to the operation of the die in that during the fabrication process, the segments were tooled to fit in an exact sequence. Deviation from this sequence results in improper assembly of the die. For this reason, each segment of the die has a numeral (1, 2, 3, or 4) engraved on its upper surface to aid in proper

and consistent assembly. For this project, the segments were always assembled in numerical order from 1 to 4 in a clockwise direction around the center hub on the baseplate. Fig. 34 shows the orientation of the segments around the center hub of the baseplate.

Once the segments of the die are assembled in the proper sequence around the hub on the baseplate the clamp ring is then placed down over the segments and bolted into place with the four high tensile strength bolts as shown in Fig. 35. The proper orientation of the clamp ring to the baseplate is important in order that the four bolts may fit properly. These bolts should never be forced into place as "stripping" of the threads on either the bolt or in the base plate may result. These bolts should only be tightened to a snug degree and should never be tightened 100 percent. From constant repetition of the die assembly procedure, the technician will develop a "feel" for the proper snugness of these bolts as the degree of tightness of these bolts directly affects the degree of friction between the die chamber and the ram.

If the die is assembled correctly, the ram should, from its own weight, slowly "glide" or lower onto the material as the air being compressed in the die chamber "escapes" through the space between the ram and the die walls. No resistance should occur from friction created between the ram and die walls. Tolerances are extremely close because it is important that there not exist a significant amount of play between the ram and the die wall. From experience, if the space between the ram and the die walls is too large, upon the addition of the compaction force, the powder material will be squeezed

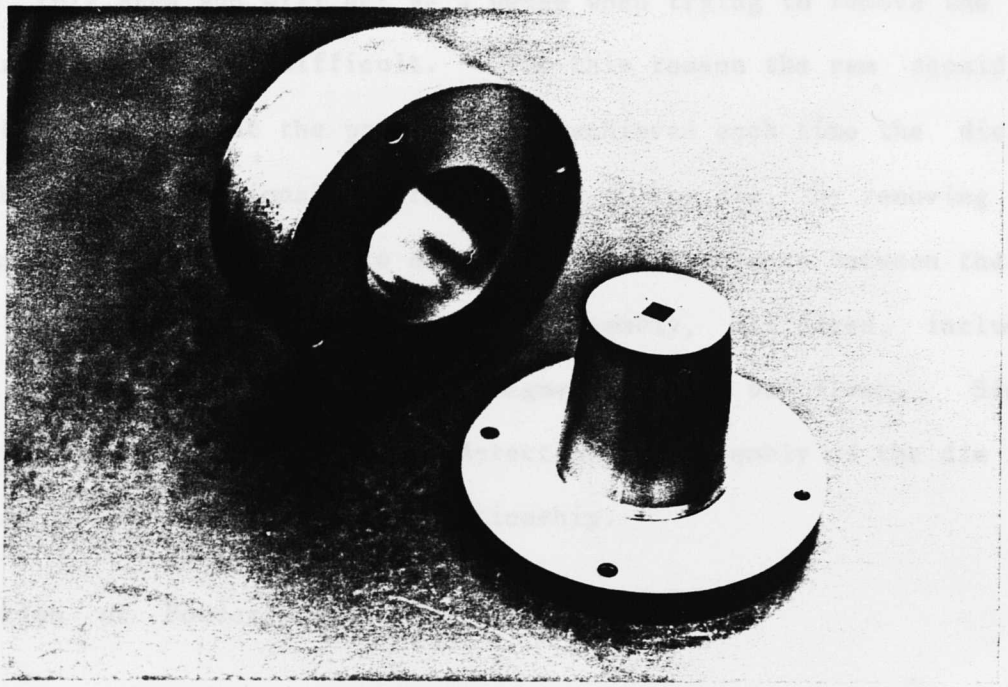

Fig. 34 - Individual die segments placed around center hub

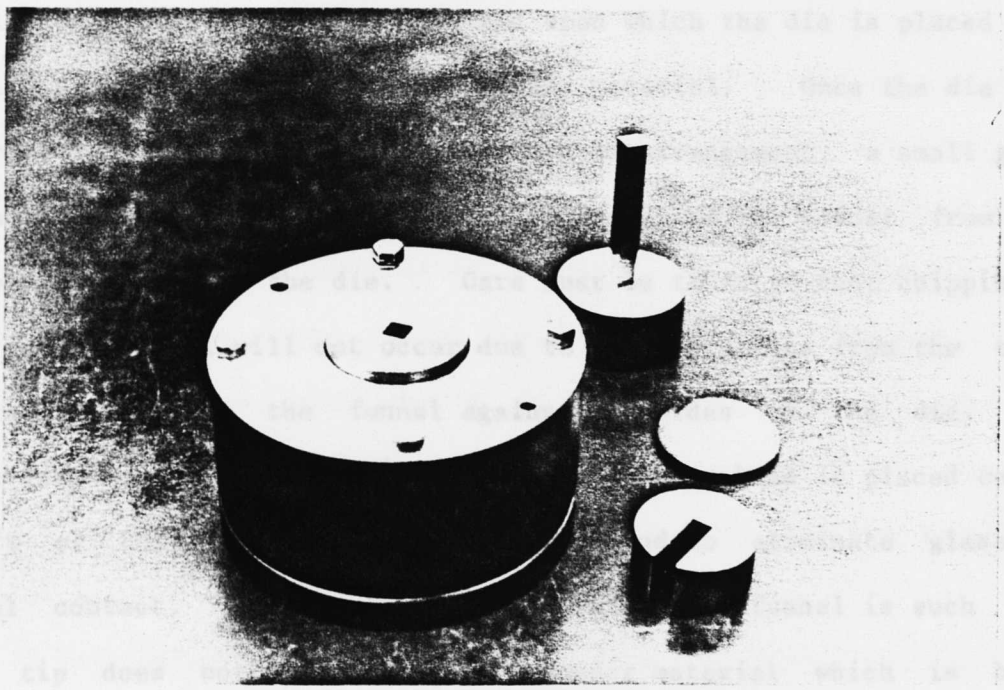

Fig. 35 - Clamp ring placed around die segments

into this area and will act as a wedge when trying to remove the ram making its removal difficult. For this reason the ram should be checked to see that the proper fit is achieved each time the die is assembled. Occasionally reassembly of the die, by removing the clamp ring, is necessary to obtain a proper clearance between the ram and the die. Also with proper die assembly, all edges, including both the top and sides of the die segments, will be flush. Simply feeling of these surfaces will detect proper assembly of the die and insure the correct die - ram relationship.

Addition of Powder to the Die

Once the die is assembled and checked the ram is removed and the powder carefully placed into the die chamber. For this process, a table or platform vibrator was used upon which the die is placed for preliminary consolidation of the powder material. Once the die and vibrating table are in the proper working arrangement, a small glass funnel is placed in the die for the transfer of the powder from the paper cup holder to the die. Care must be taken so that chipping of the glass funnel will not occur due to the vibrations from the table vibrator rattling the funnel against the sides of the die. To alleviate some of this problem, a small rubber band is placed on the shaft of the funnel to act as a cushion and to eliminate glass to metal contact. The shaft length of this glass funnel is such that the tip does not extend into the powder material which is being introduced.

As shown in Fig. 36, the paper cup is squeezed between the thumb

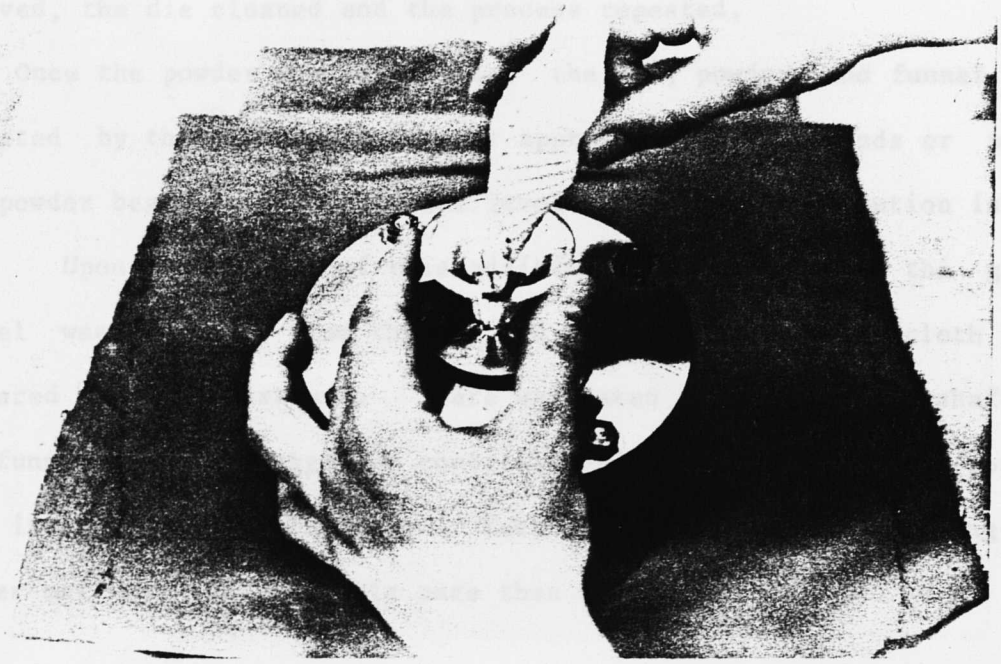

Fig. 36 - Powder being placed in the die

and forefinger of one hand with the middle finger providing support to the bottom of the cup. The thumb and middle finger of the other hand are used to rotate the funnel as the powder is gently tapped from the paper cup into the mouth of the funnel. This provides an even placement of the powder in the die. The powder is placed in the funnel slowly and continuously in such a way that it does not accumulate at the neck of the funnel. Light brushing of the paper cup with a small fine bristled brush while it is held over the funnel is used as a means of removing all of the powder.

If any powder is lost outside of the die during this process, the resulting porosity of the compact would be greater than desired. Therefore if powder is lost during this process, the powder must be

removed, the die cleaned and the process repeated.

Once the powder was in the die, the die, powder, and funnel were vibrated by the table vibrator for approximately 15 seconds or until the powder began to flow into one level of uniform distribution in the die. Upon completion of this additional vibrating time the glass funnel was removed from the die and wiped clean with a cloth and prepared for the next use. Care was taken to see that the shaft of the funnel was not clogged or containing significant amounts of powder upon its removal from the die. Each paper cup holder containing the powder material was used only once then discarded.

Compaction of the Powder

Once the powder has been properly added to the die, the ram is inserted into the die as shown in Fig. 37. The symmetric construction of the ram and die allows for placement of the ram in the die chamber in any direction. The ram is inserted slowly into the die so that the amount of powder material which is lost with the escaping compressed air is small. No lubricant of any kind was used on the ram or die walls during the compaction process.

Once the ram is properly placed in the die, the spacer piece is then placed around the ram as shown in Fig. 38. The purpose of this spacer piece, as discussed in Chapter III, is to produce a constant nominal compact volume at full compaction and provide for manual control over the amount of compaction force which is applied to the powder material. This is accomplished by the continuous movement of

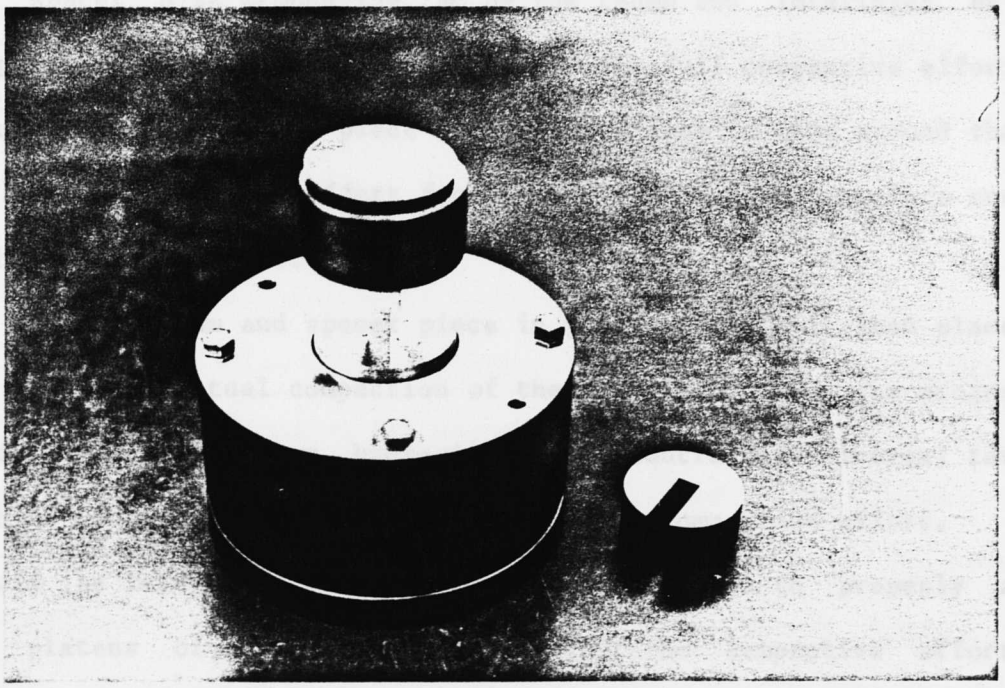

Fig. 37 - Compaction ram placed in the die

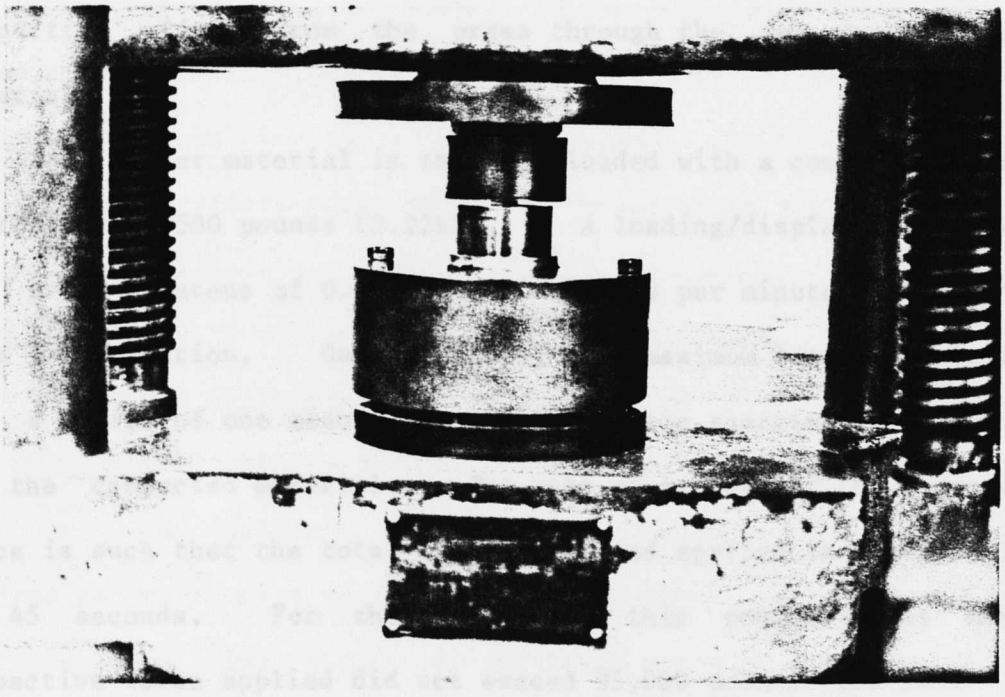

Fig. 38 - Assembled die in press awaiting application of compaction effort

the spacer disk around the ram by the thumb and forefinger as the compactive force is being applied. The full compactive effort is reached when the spacer piece is no longer free to move around the ram due to the compactive effort forcing the ram head to come into contact with the spacer piece.

With the ram and spacer piece in place, the die is then placed in the press for actual compaction of the material. For the project at hand, a Southwark-Emery hydraulic, speed controlled universal testing machine was used for the application of the compaction effort. Care should be taken when placing the die in the press to properly align the platens of the press in order that the compactive effort is uniformly applied. From Fig. 37 and Fig. 38, the die also has a spherical seat and washer which allows for the proper distribution of compactive effort from the press through the ram to the powder material.

The powder material is then pre-loaded with a compactive load of approximately 500 pounds (2.22kN). A loading/displacement rate of the press platens of 0.05 inches (1.27 mm) per minute was used for this investigation. Once obtained, the maximum load was maintained for a period of one minute due to the elastic energies which develop in the compacted material. The rate of release of the compaction force is such that the total compactive load applied is released in 30 to 45 seconds. For the purposes of this project, the maximum compactive force applied did not exceed 35,000 pounds (156 kN). The production pressure is determined by dividing the production force which the press registered by the cross-sectional area of the ram.

This cross-sectional area was determined to be 0.1561 square inches (1.0074 square centimeters).

Removal of the Compact

Once the compactive force is released, the die is removed from the pressing machine and the prepared powder compact readied for the curing stage. To remove the prepared compact from the die, the first step is the removal of the spacer piece followed by the removal of the ram from the die. The removal of the ram from the die is accomplished by slowly lifting the ram from the die by hand. Care should be taken so that the prepared compact is not damaged by the vacuum created in the die when the ram is removed.

The next step is the removal of the four bolts which hold the clamp ring in place. Once these bolts are removed, the puller bar is assembled across the top of the die segments as shown in Fig. 39. Due to the extremely high compaction forces which are used, the die segments experience some degree of radial displacement which causes them to become firmly wedged into the clamp ring. For this reason the puller bar is required to "break down" the die. The clamp ring is freed by tightening the two bolts on the puller bar as shown in Fig. 39. By tightening these bolts, a vertical lifting force is applied to the clamp ring while the die segments are held in place.

Once free, the clamp ring is then removed by hand and the four interior segments of the die were peeled carefully away from the prepared compact sitting on the center hub as shown in Fig. 40.

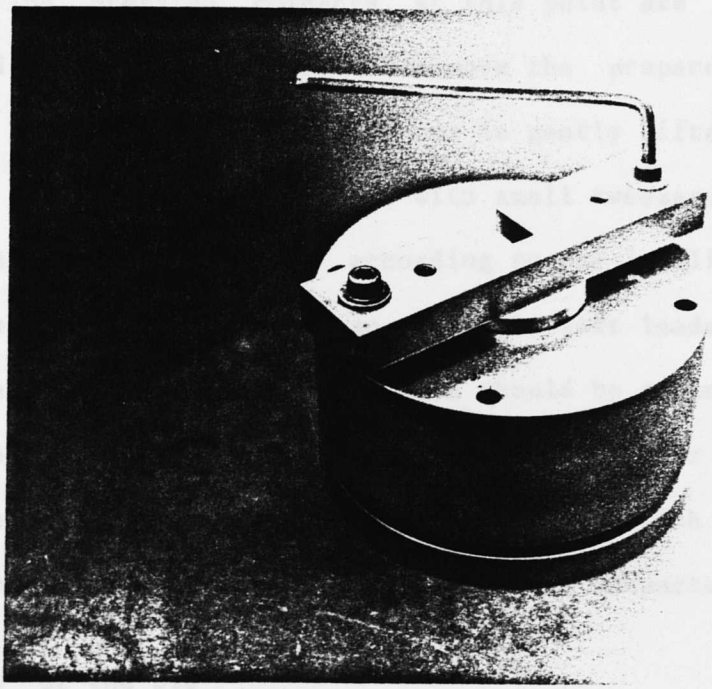

Fig. 39 - Puller bar assembled across the top of the die segments

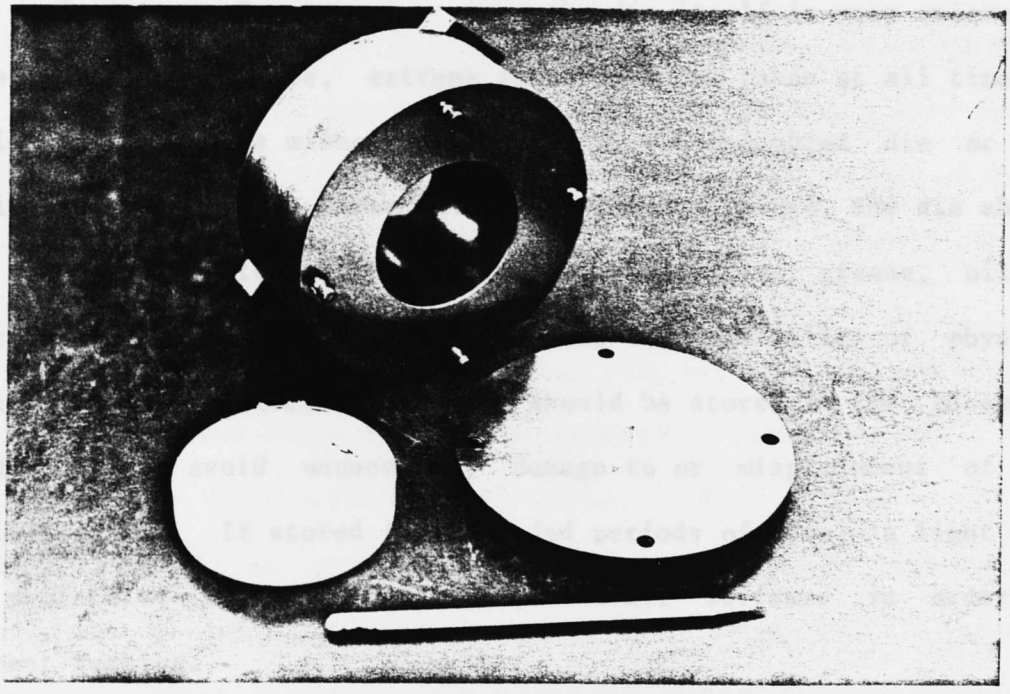

Fig. 40 - Disassembled die with prepared compact on center hub

The prepared compacts at this point are stable enough to be handled gently by hand. To remove the prepared compact from the hub on the base plate, the compact is gently lifted from the hub between the thumb and forefinger or with small tweezers. The compact is then labeled carefully, according to the labeling system discussed in Chapter III, by using an extremely soft leaded pencil on opposite sides of the compact. Care should be taken when numbering the compacts to not scratch any powder from their surfaces.
Once numbered, the compact is then placed with the aid of tweezers in a holding container until the remaining compacts have been prepared.

Care of the Die

At this point it is appropriate to discuss the proper care of the die equipment. In general, since the die itself is very unique and expensive to fabricate, extreme care should be taken at all times to avoid the careless mishandling of either the assembled die or the individual pieces when disassembled. When not in use, the die should be stored in a clean environment where exposure to grease, oil or particulate material is not possible and the possibility of physical damage is also limited. The die should be stored in the assembled condition to avoid unnecessary damage to or misplacement of the various parts. If stored for extended periods of time, a light coat of protective oil should be applied to all surfaces in order to prevent rusting.

Upon the removal of the die from storage and the actual preparation of compacts, all surfaces should be wiped clean of oil

using a clean cloth and liquid solvent cleaning material such as methyl ethyl ketone or isopropyl alcohol. Upon cleaning, each individual piece should be placed on a clean cloth until all pieces are clean and the die is to be assembled. In addition to this initial cleaning of the die for use, complete disassembly and cleaning of all pieces should be performed upon the preparation of each and every compact. This is necessary to avoid the accumulation of powder material on the tooled portions of the die pieces which will hinder proper die assembly and operation.

Processing of the Prepared Compacts

Upon completion of preparation of the desired number of compacts, the freshly prepared compacts are then weighed and measured before commencement of curing. For all compacts prepared, measuring should be performed first followed by weighing. In this way a more correct initial weight value is determined because the small amount of powder which is generally scratched from the surface of the compact in the process of measuring is taken into account.

For this project, as shown in Fig. 41, measuring of the compacts was performed with a the aid of a micrometer to 0.0001 inches. Later conversion to centimeters was required. Each dimension of the cubicle compacts was measured three times from which an average was determined as the value of that particular dimension. Generally each of the three readings for a particular dimension were taken from a different location on the surface measured.

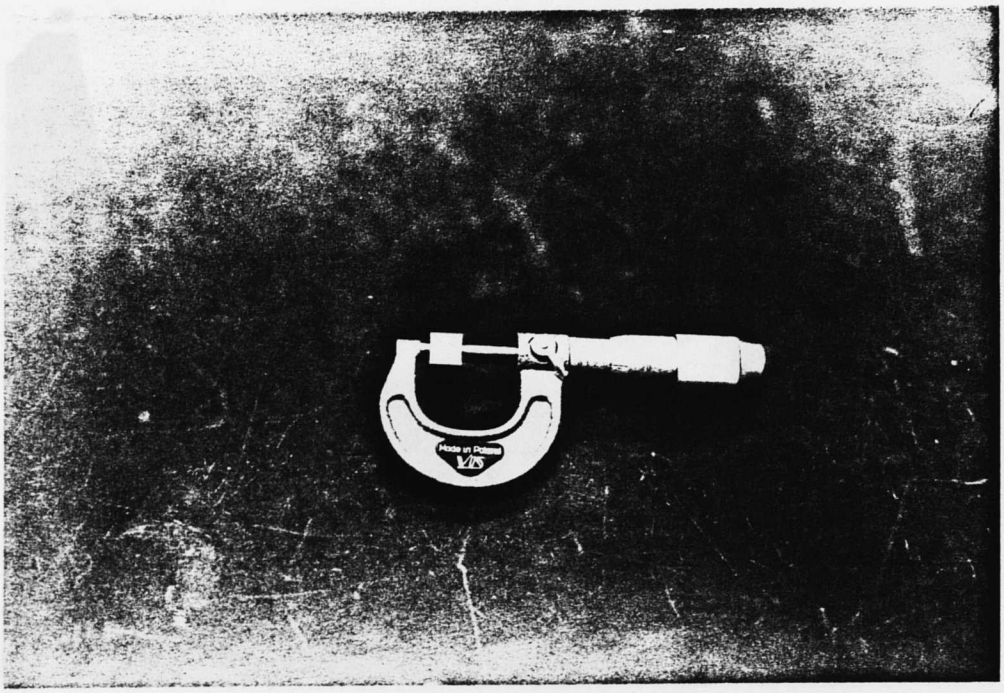

Fig. 41 - Compact and micrometer for dimension determination

As shown in Fig. 42, weighing of the prepared compacts was performed as mentioned earlier. Unlike the determination of the dimensions for the compacts, only one weighing was made for each compact. As discussed, this measurement was taken after the labeling of the compact and also the measuring process; two procedures which caused minor loss of powder from the surfaces of the compacts.

The Curing Regime

Following the measuring and weighing of the compacts, the freshly prepared compacts were then ready to begin the curing process for the desired duration. The fresh compacts, in an open container , were initially placed in a controlled atomosphere of 73 F (22.8 C) at 100

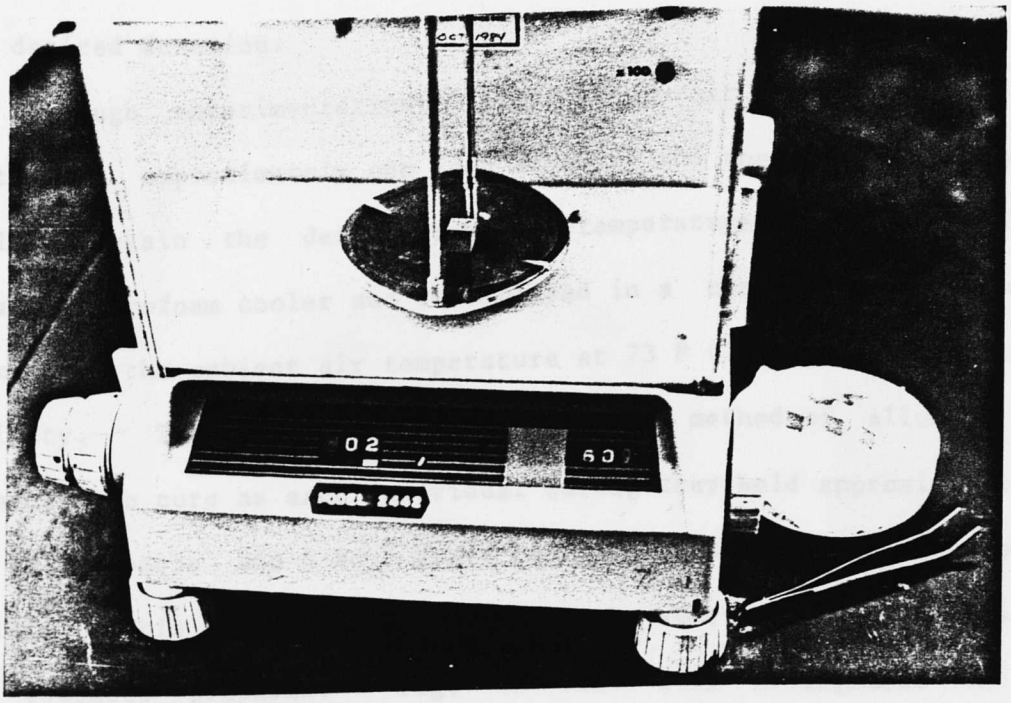

Fig. 42 - Compacts and scale for weight determination

percent humidity for a period of approximately 30 minutes during which time the compacts became acclimated to an abundance of moisture for hydration. During this procedure, the container holding the compacts would gradually collect water in the 30 minutes of time at which point the compacts were effectively immersed in water. The time at initial placement of the compacts in the 100 percent humidity environment was recorded as the beginning of the curing period.

The compacts were then removed from this initial curing exposure condition and placed in the main curing regime for the remainder of the desired curing period. The main curing regime consisted of immersing the prepared compacts in a supersaturated lime and distilled water solution at a constant temperature of 70 F (21 +/- 1/2 C) for

the desired duration.

Through experimentation, it was found that an open curing pan containing approximately one inch of water and the hydrating compacts would maintain the desired constant temperature when placed in a closed styrofoam cooler and then placed in a controlled atomosphere room with the ambient air temperature at 73 F (22.8 C) and 50 percent humidity. This procedure was an excellent method of allowing the compacts to cure as each individual curing tray held approximately 50 to 60 compacts and 4 to 5 trays could be placed in one styrofoam cooler. This allowed for essentially the same curing conditions for all compacts prepared. Fig. 43 shows this arrangement in the styrofoam cooler which inturn was covered and placed in the controlled atomosphere condition.

There is one point which should be made concerning the curing regime used. The pans which contained the hydrating compacts as mentioned were not covered. This allowed for evaporation of water and the increasing of the concentration of the lime in the remaining solution in the curing pan. It is suggested because of this action that closed or covered curing containers be used rather than the open containers.

Compressive Testing of the Compacts

Once the proper curing period was achieved, the final stage of the laboratory investigation was the compressive testing to failure of the cured compacts. To accomplish this an Instron servo-mechanical,

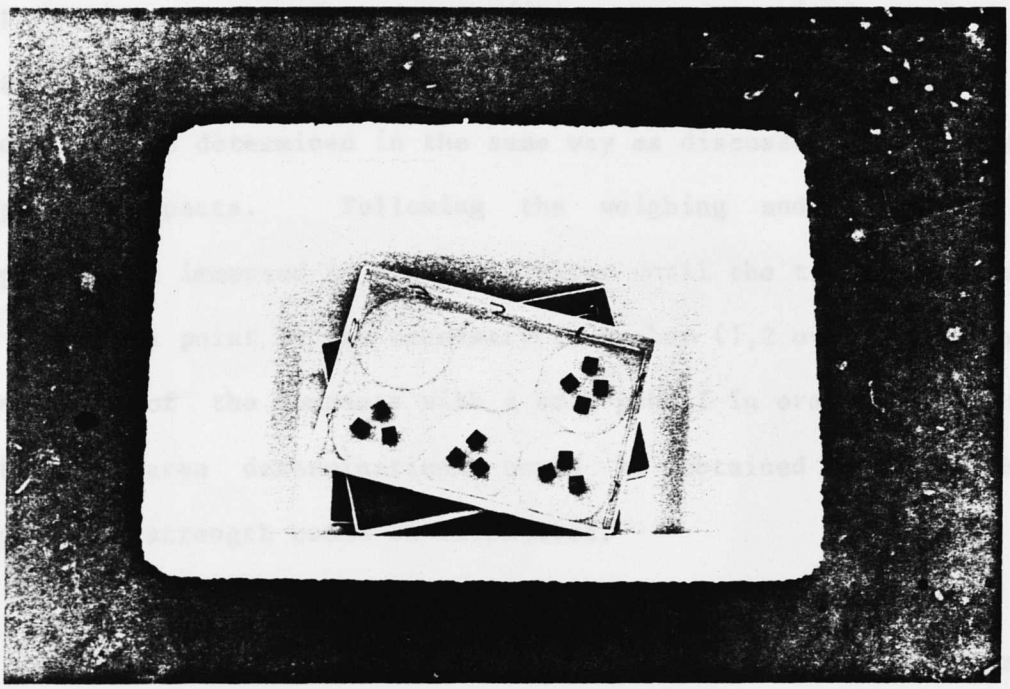

Fig. 43 - Compact curing arrangement

speed controlled universal testing machine was utilized. The rate of loading was 0.01 inches (.25 mm) per minute.

As discussed in the earlier chapters, cured compacts were tested to failure in both a wet surface dry condition and an oven dried condition. Because of these conditions, the actual testing process was also different for each type of compact tested.

For compacts tested in the wet surface dry condition, upon completion of the desired curing period, the compacts were removed from the curing solution within one hour of the time of placement in the curing solution. Following the removal of lime deposits, by gently wiping or brushing the surface, which had collected on the surface of the compacts, a post curing weight and measurement of the

compact in a wet surface dry condition was determined. Both the weight and dimensions of the cured compact in the wet surface dry condition were determined in the same way as discussed for the freshly prepared compacts. Following the weighing and measuring, the compacts were immersed in distilled water until the time of failure.

At this point it was necessary to number (1,2 or 3) the various dimensions of the compacts with a soft pencil in order that cross-sectional area determinations could be obtained from which a compressive strength could be calculated.

At the time of failure, the compact to be tested was removed from the water and again towel dried to a condition of wet surface dried. Particular attention was devoted to drying the surfaces that would be in contact with the platens. Once the proper condition was achieved, the compact was placed on the lower platen of the testing equipment, as shown in Fig. 44, and preloaded to approximately 500 pounds (2.22 kN). Once a load of 1,000 pounds (4.45 kN) was attained, distilled water was applied to the four exposed sides of the compact with the aid of a squirt bottle. The purpose of this was to keep the compact surfaces from drying to a large degree and creating strength producing restrained shrinkage stresses and thereby causing a false strength determination. Upon failure, the compact remains were collected and stored in a sealed plastic bag for future use.

It should be pointed out at this point that in the failure of the compacts, the application of the failure loading was <u>not</u> the same axis of the cubicle compact for which the compactive load was applied in

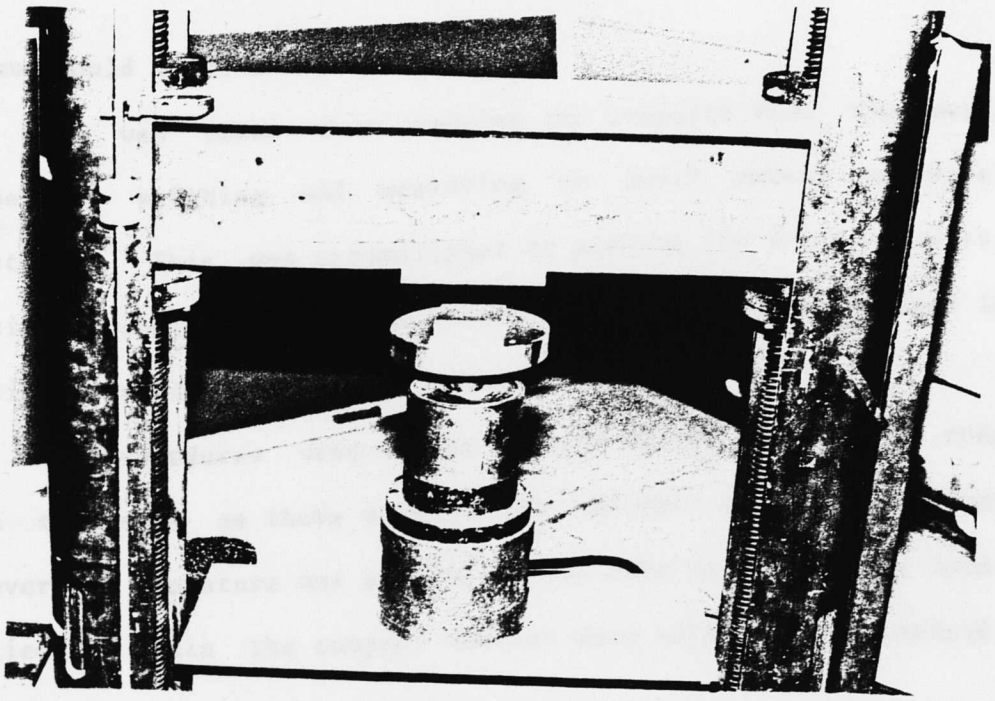

Fig. 44 - Apparatus used for the failure of the compacts

its preparation. This was done to avoid undue influences caused by denser portions of the compacts on the compressive strength.

For the compacts tested in the oven dried condition, upon the completion of curing, the compact was weighed and measured in the wet surface dry condition as before. However upon completion of the weighing and measuring of all the compacts, they were placed in a convection oven at a temperature of 220 F (105 C) for 48 hours of drying. To support the fact that 48 hours of drying was an adequate period of time to eliminate all free water, the compacts were weighed at 24 and 42 hours of oven drying in addition to 48 hours. Upon completion of the 48 hour duration, the compacts were also measured as described earlier three times for each dimension so that an oven dried

volume could be determined.

Care was taken when removing the compacts from the oven and subsequent weighing and measuring to avoid excess exposure to moisture. This was accomplished by placing the dried compacts in a dessicator containing calcium sulfate. The compacts remained in the dessicator until the time of failure.

The procedures used in the failure of the oven dried compacts were the same as those employed for the wet surface dry compacts however no moisture was applied to the cube as the failure load was applied. Again the compact remains were collected and stored for future use.

APPENDIX C

POWDER WEIGHT DETERMINATION PROCEDURES

Introduction

The purpose of this Appendix is to cover in detail the procedure for determining the amount of powder material needed to produce a desired porosity. As mentioned in the report, due to the principle on which the die operates, the amount of powder which is placed in the die chamber controls the resulting initial porosity. From laboratory experimentation, the relationship between the two was found to be linear in nature. Because of this linear relationship, the determination and use of a simple factor which accounted for the variation in specific gravity of the fly ash material and the amount of fly ash in the compact was possible.

Laboratory Results

To determine the initial relationship between the weight of powder material used and the porosity obtained, ten experimental trial series consisting of three to eight samples each were prepared from the Type III anhydrous cement powder. The results of these trials are listed in Table 2.

The calculated initial porosities shown in this table were determined with the following equation:

$$P_i = 1 - ((W_t / V_t)((SG_c)(\%C) + (SG_{fa})(\%FA))) \qquad (1)$$

where: P_i is the calculated initial porosity,

W_t is the total weight of the prepared compact,

V_t is the total volume of the prepared compact,

SG_c is the specific gravity of the cement,

SG_{fa} is the specific gravity of the fly ash,

%C is the percentage of the powder used for the compact which is cement, and

%FA is the percentage of the powder used for the compact which is fly ash.

Using the calculated initial porosity and powder weight values shown in Table 2, a linear regression analysis was performed to determine if a linear relationship existed between the two paramters. This analysis provided the following equation:

$$\text{Porosity} = -17.8013\ (\text{Powder Weight}) + 71.2672 \qquad (2)$$

where :

Porosity refers to the calculated initial porosity, and

Powder weight refers to the weight of anhydrous cement powder material which was placed in the die for compaction. Powder weight is not the weight of the compact after compaction.

The coefficient of determination (r^2) for this equation was 0.994.

Because of the excellent relationship which existed between the initial powder weight and the calculated initial porosity for the

Table 2 - Preliminary data used to determine the relationship between powder weight and calculated initial porosity

Series No.	No. of Samples	Powder Weight (g)	Initial Density (g/cm^3)	Calculated Initial Porosity (percent)
P3	6	2.5386	2.3145	26.05
P4	8	2.7290	2.4136	22.89
P5	6	2.8469	2.4723	21.02
P7	6	2.4673	2.2814	27.11
P8	6	2.5939	2.3508	24.89
P9	3	2.3250	2.1870	30.13
P10	4	2.6500	2.3875	23.72
P11	3	2.3750	2.2186	29.12
P12	4	2.4200	2.2458	28.25
P13	4	2.7500	2.4392	22.07

anhydrous cement powder, this equation was used to determine the powder weights needed to produce a desired initial porosity condition for the various percentages of fly ash replacement of the cement. Due to the nominal volume principle on which the die operates, with the replacement of a certain amount of cement powder with fly ash material of lower specific gravity, the relationship exists whereby less powder material by weight will be required to produce the same porosity level.

For example, if 2.8700 grams of anhydrous cement material produces a porosity of 20 percent, the values of %C and %FA in Eq. (1) are 1.0 and 0.0 respectively. The specific gravity value is entirely that of the cement powder. If fly ash is introduced into the system, the specific gravity used to calculate the porosity is a proportional

specific gravity depending on the percentages of fly ash and cement in the system. In this respect then, with all factors such as volume and porosity held constant, the control weight of cement only to produce a desired porosity may be adjusted by multiplying a factor determined by:

$$((SG_c)(\%C) + (SG_{fa})(\%FA))/SG_c \qquad (3)$$

to determine the powder weight needed of a material containing a percentage of fly ash.

From these considerations, the following factors were determined for the different percentages of fly ash as shown.

Percent Fly Ash	Calculations		Factor
0% Fly Ash	(Cement Only Control)	=	1.0000
10% Fly Ash	((3.13x0.9)+(2.75x0.1))/3.13	=	0.9877
20% Fly Ash	((3.13x0.8)+(2.75x0.2))/3.13	=	0.9754
30% Fly Ash	((3.13x0.7)+(2.75x0.3))/3.13	=	0.9631

The cement specific gravity value used was 3.13 and the fly ash specific gravity value for the sieved material was 2.75.

Once the factors were determined, the next step was the calculation of the actual powder weights for the various percentages of fly ash replacements. From this procedure, the values as shown in Table 3 were determined and utilized throughout the remainder of the project as the weight of powder material placed in the die to achieve

Table 3 - Initial powder weights used to produce desired calculated initial porosities

Desired Initial Porosity (percent)	Powder Weights Used (grams)			
	Percent Fly Ash Replacement for Cement (percent)			
	0	10	20	30
	Factor = 1.0000	Factor = 0.9877	Factor = 0.9754	Factor = 0.9631
20	2.8700	2.8347	2.7994	2.7641
23	2.7125	2.6791	2.6458	2.6124
26	2.5425	2.5112	2.4800	2.4487
29	2.3750	2.3458	2.3166	2.2874
32	2.2100	2.1828	2.1556	2.1285

a desired initial porosity condition.

It is important to note that at the time these factors were initially determined, Eq. (1) was developed from more of an intuitive approach by considering the relationships between porosity, density, specific gravity, percent cement and percent fly ash. However through subsequent correspondence with Dr. Matti Relis, the actual mathematical derivation of the relationship between porosity, density, specific gravity, percent cement, and percent fly ash produced a different equation. This derived equation is developed in Appendix D.

From the laboratory results however, it appears that the considerations discussed in this appendix are adequate for determining quantities of powder containing various percentages of fly ash to be used in producing a desired target initial porosity. For actual initial porosity calculations, the derived equation of Appendix D is the correct mathematical relationship and was used for determining the calculated initial porosity content of the compacts prepared for this investigation.

APPENDIX D

CALCULATED INITIAL POROSITY FORMULA DERIVATION

Introduction

The calculated initial porosity is a measure of the volume of a prepared compact which is void space before exposure to moisture and subsequent hydration occurs. The determination of this value is dependent on the specific gravity of the material, the volume, and the weight of the prepared compact. This Appendix contains the derivation of the relationship between these variables.

One Component System

A one component system refers to the use of only one type of powder material for the compact preparation. In this case, the Type III cement powder would be the material. The derivation of the calculated intial porosity equation for this condition follows.

Initially the compact has a total volume consisting of the volume of cement (V_c) and the volume of the voids between the cement particles (V_{air}). The total weight (W_t) of the compact is the weight of cement powder (W_c) composing the compact. In equation form, these relationships are:

$$V_t = V_c + V_{air} \tag{1}$$

$$W_t = W_c \tag{2}$$

Dividing Eq. (1) by the total volume (V_t) of the compact, the equation becomes:

$$1 = V_c/V_t + V_{air}/V_t \qquad (3)$$

The ratio of V_a/V_t is defined as the fraction of the total compact volume which is void space or porosity (P_i). Eq. (3) can then be written as:

$$1 = V_c/V_t + P_i \qquad (4)$$

Using the relationship of specific gravity, the volume of cement (V_c) can be written as:

$$V_c = W_t/SG_c \qquad (5)$$

where SG_c is the specific gravity of the cement material. Eq. (4) then becomes:

$$1 = (W_t/SG_c)(1/V_t) + P_i \qquad (6)$$

Solving for P_i, the equation then becomes:

$$P_i = (1-(W_t/V_t)(1/SG_c)) \times 100 \qquad (7)$$

where P_i is the initial porosity of the compact on a percentage basis. Realizing that W_t/V_t is the density of the prepared compact, the calculation of the initial porosity for one component system is accomplished.

Two Component System

For a two component system whereby two materials with different specific gravity values are combined in various percentages, the determination of the calculated initial porosity becomes more complex.

Initially the volume of a prepared compact, in the case of this work, is the volume of cement, the volume of fly ash and the volume of void space. In equation form this relationship is:

$$V_t = V_c + V_{fa} + V_{air} \tag{8}$$

Dividing through by the total volume V_t, the equation becomes:

$$1 = V_c/V_t + V_{fa}/V_t + V_{air}/V_t \tag{9}$$

Again the ratio V_{air}/V_t can be defined as the initial porosity (P_i) or void space as a fraction of the total volume. Eq. (9) then becomes:

$$P_i = 1 - (V_c + V_{fa})/V_t \tag{10}$$

Concerning the weight relationship in a two component prepared compact, the total weight (W_t) equals the weight of the fly ash (W_{fa}) plus the weight of the cement material (W_c). In equation form this becomes:

$$W_t = W_c + W_{fa} \tag{11}$$

Dividing through by the total weight (W_t), the equation becomes:

$$1 = W_c/W_t + W_{fa}/W_t \tag{12}$$

from which the ratios of the weights of the individual component materials may be defined as:

$$\%C = W_c/W_t \text{ and } \%FA = W_{fa}/W_t \tag{13}$$

Also using the specific gravity of each of the inidividual components, the volumes of cement and fly ash can be written as:

$$V_c = W_c/SG_c \tag{14}$$

$$V_{fa} = W_{fa}/SG_{fa} \tag{15}$$

Eq. (10) then becomes:

$$P_i = 1 - (1/V_t)(W_c/SG_c + W_{fa}/SG_{fa}) \qquad (16)$$

However this equation is impractical in that only the final density of the prepared compacts is known and not the individual weight values for each of the two powders. In order to have a more usable equation the following modification is made:

$$P_i = 1 - (W_t/V_t)((1/SG_c)(W_c/W_t) + (1/SG_{fa})(W_{fa}/W_t)) \qquad (17)$$

As defined by the equations of (13), W_c/W_t and W_{fa}/W_t are the known percentages of cement (%C) and fly ash (%FA) by weight in the mix used to prepare the compact. From this consideration then, the calculated initial porosity of the two component anhydrous system may be determined by:

$$P_i = (1 - ((W_t/V_t)(\%C/SG_c + \%FA/SG_{fa}))) \times 100 \qquad (18)$$

where: P_i is the calculated initial porosity,

W_t/V_t is the density of the prepared compact,

%C is the percentage of cement by weight in the mixed powder used for the compact,

%FA is the percentage of fly ash by weight in the mixed powder used for the compact,

SG_c is the specific gravity of the cement, and

SG_{fa} is the specific gravity of the fly ash material.

APPENDIX E

LABORATORY DATA

Table 4 - Laboratory data for SSD compacts containing 100 percent portland cement and cured for three days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)	Calc. Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
B11	$\bar{X}$ =	182,000	1,255	2.8684	1.1477	2.4992	20.15	3.1252	1.1567	2.7019	10.51	44,300[c]	305[c]
	σ =	3700	25	0.0006	0.0012	0.0027	0.08	0.0011	0.0012	0.0030		800	6
B12	$\bar{X}$ =	121,000	834	2.7093	1.1191	2.4209	22.65	2.9894	1.1277	2.6510	13.00	40,500[b]	279[b]
	σ =	3000	19	0.0007	0.0012	0.0028	0.08	0.0013	0.0018	0.0037		1000	7
B13	$\bar{X}$ =	70,300	485	2.5382	1.0910	2.3266	25.67	2.8513	1.0971	2.5989	16.01	39,500[b]	272[b]
	σ =	3100	22	0.0013	0.001	0.002	0.06	0.0018	0.0012	0.0023		1900	13
B14	$\bar{X}$ =	35,400	244	2.3714	1.0679	2.2206	29.05	2.7195	1.0736	2.5329	19.38	37,100[c]	256[c]
	σ =	1400	9	0.0003	0.001	0.0021	0.06	0.0008	0.0011	0.0027		1700	12
B15	$\bar{X}$ =	14,200	98	2.2083	1.0516	2.0998	32.91	2.5930	1.0569	2.4534	23.23	35,000[b]	242[b]
	σ =	300	2	0.0005	0.001	0.0019	0.05	0.0018	0.0016	0.0029		500	3

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7

Table 5 - Predrying laboratory data for OD compacts containing 100 percent portland cement and cured for three days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)
b11	$\bar{X}$ =	174,200	1,201	2.8670	1.1468	2.4999	20.13	3.1282	1.1549	2.7087
	σ =	600	4	0.002	0.0015	0.0023	0.07	0.0022	0.0015	0.0028
b12	$\bar{X}$ =	117,800	812	2.7069	1.1174	2.4226	22.60	2.9901	1.1240	2.6603
	σ =	2700	19	0.0026	0.0008	0.0018	0.05	0.0022	0.0011	0.0037
b13	$\bar{X}$ =	68,800	475	2.5406	1.0911	2.3285	25.61	2.8561	1.0982	2.6007
	σ =	2000	14	0.0006	0.0015	0.0032	0.09	0.0017	0.0019	0.0038
b14	$\bar{X}$ =	33,300	230	2.3718	1.0674	2.2222	29.00	2.7203	1.0732	2.5349
	σ =	1100	8	0.0004	0.0007	0.0015	0.04	0.0011	0.0013	0.0033
b15	$\bar{X}$ =	14,400	99	2.2064	1.0500	2.1013	32.87	2.5940	1.0556	2.4575
	σ =	300	2	0.0024	0.0007	0.0031	0.09	0.0014	0.0011	0.0034

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation

Table 6 - Postdrying laboratory data for OD compacts containing 100 percent portland cement and cured for three days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		24 Hour OD Weight (g)	42 Hour OD Weight (g)	48 Hour OD Weight (g)	48 Hour OD Volume (cm^3)	48 Hour OD Density (g/cm^3)	Measured Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
b11	$\bar{X}$ =	3.0150	3.0069	3.0079	1.1505	2.6145	10.42	64,900[b]	448[b]
	σ =	0.0020	0.0019	0.0019	0.0013	0.0031		2000	14
b12	$\bar{X}$ =	2.8554	2.8468	2.8467	1.1178	2.5468	12.76	61,600[c]	424[c]
	σ =	0.0025	0.0027	0.0028	0.0009	0.0036		1400	10
b13	$\bar{X}$ =	2.6865	2.6799	2.6780	1.0899	2.4571	16.22	55,300[b]	381[b]
	σ =	0.0009	0.0008	0.0008	0.002	0.0046		1500	10
b14	$\bar{X}$ =	2.5110	2.5119	2.5103	1.0631	2.3613	19.57	48,900[b]	337[b]
	σ =	0.0004	0.0004	0.0005	0.001	0.0021		1700	12
b15	$\bar{X}$ =	2.3523	2.3539	2.3517	1.0438	2.2530	22.95	42,900[b]	296[b]
	σ =	0.0026	0.0025	0.0026	0.0008	0.0035		1600	11

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7

Table 7 - Laboratory data for SSD compacts containing 100 percent portland cement and cured for seven days (sample size = 8 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)	Calc. Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
C11R $\bar{X}$ =	182,900	1,261	2.8672	1.1468	2.5001	20.13	3.1304	1.1572	2.7052	10.64	49,000	338
σ =	3600	25	0.0006	0.001	0.0024	0.07	0.0016	0.0013	0.0022		1700	12
C12R $\bar{X}$ =	122,600	845	2.7089	1.1160	2.4272	22.45	2.9896	1.1240	2.6599	12.65	48,000[b]	331[b]
σ =	2,700	19	0.0022	0.0006	0.0014	0.04	0.0022	0.0019	0.0046		600	4
C13R $\bar{X}$ =	70,700	487	2.5382	1.0896	2.3293	25.58	2.8505	1.0967	2.5991	15.36	45,000[b]	310[b]
σ =	3,700	26	0.0032	0.0011	0.0026	0.07	0.0027	0.0014	0.0024		400	3
C14R $\bar{X}$ =	33,300	229	2.3725	1.0679	2.2217	29.02	2.7208	1.0735	2.5345	18.34	44,900[b]	310[b]
σ =	1,000	7	0.0003	0.0011	0.0023	0.07	0.0011	0.0010	0.0018		900	6
C15R $\bar{X}$ =	13,800	95	2.2073	1.0506	2.1010	32.87	2.5956	1.0574	2.4547	21.68	41,900[c]	289[c]
σ =	500	3	0.0003	0.0005	0.0011	0.03	0.0007	0.0012	0.0024		400	3

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7

Table 8 - Predrying laboratory data for OD compacts containing 100 percent portland cement and cured for seven days (sample size = 8 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)
c11r $\bar{X}$ =	183,600	1,266	2.8680	1.1474	2.4996	20.14	3.1339	1.1587	2.7047
c11r σ =	3,000	20	0.0006	0.0005	0.0011	0.03	0.001	0.0015	0.0029
c12r $\bar{X}$ =	124,000	855	2.7099	1.1186	2.4226	22.60	2.9947	1.1251	2.6618
c12r σ =	2,000	14	0.0003	0.0012	0.0025	0.07	0.001	0.0007	0.0011
c13r $\bar{X}$ =	72,000	496	2.5388	1.0899	2.3294	25.58	2.8552	1.0975	2.6016
c13r σ =	1,200	8	0.0035	0.0009	0.0023	0.07	0.0028	0.0011	0.0027
c14r $\bar{X}$ =	35,700	246	2.3724	1.0680	2.2214	29.03	2.7222	1.0744	2.5337
c14r σ =	1,000	7	0.0004	0.0009	0.0020	0.06	0.0017	0.0008	0.0019
c15r $\bar{X}$ =	14,600	101	2.2065	1.0505	2.1005	32.89	2.5948	1.0569	2.4550
c15r σ =	500	3	0.0027	0.0007	0.0024	0.07	0.0021	0.0006	0.002

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation

Table 9 - Postdrying laboratory data for OD compacts containing 100 percent portland cement and cured for seven days (sample size = 8 unless otherwise noted)

Series and Statistic[a]	24 Hour OD Weight (g)	42 Hour OD Weight (g)	48 Hour OD Weight (g)	48 Hour OD Volume (cm^3)	48 Hour OD Density (g/cm^3)	Measured Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
c11r $\bar{X}$ =	3.0129	3.0087	3.0084	1.1552	2.6042	10.83	67,900[b]	468[b]
σ =	0.0011	0.0010	0.0010	0.0011	0.0023		1,100	8
c12r $\bar{X}$ =	2.8619	2.8538	2.8529	1.1205	2.5460	12.60	62,400[b]	430[b]
σ =	0.0016	0.0017	0.0015	0.0009	0.0025		2,300	16
c13r $\bar{X}$ =	2.6949	2.6874	2.6876	1.0910	2.4634	15.27	60,800[b]	419[b]
σ =	0.0040	0.0049	0.0041	0.0015	0.0046		1,300	9
c14r $\bar{X}$ =	2.5289	2.5248	2.5248	1.0656	2.3694	18.37	50,900[c]	351[c]
σ =	0.0028	0.0026	0.0025	0.0005	0.0026		2,200	15
c15r $\bar{X}$ =	2.3672	2.3646	2.3649	1.0466	2.2595	21.75	47,700[c]	329[c]
σ =	0.0029	0.0029	0.0029	0.0007	0.0018		1,300	9

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7

Table 10 - Laboratory data for SSD compacts containing 100 percent portland cement and cured for twenty-eight days (sample size = 8 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)	Calc. Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
D11R $\bar{X}$ =	184,300	1,271	2.8668	1.1470	2.4994	20.15	3.1321	1.1590	2.7025	10.10	49,400[b]	341[b]
σ =	3,100	21	0.0010	0.0007	0.0019	0.05	0.0014	0.0012	0.0026		3,000	21
D12R $\bar{X}$ =	125,100	863	2.7091	1.1186	2.4219	22.62	2.9975	1.1278	2.6579	12.35	49,700[b]	342[b]
σ =	1,800	12	0.0006	0.0006	0.0013	0.04	0.0005	0.0011	0.0026		700	5
D13R $\bar{X}$ =	72,700	501	2.5390	1.0913	2.3267	25.67	2.8570	1.0981	2.6017	15.14	48,300[b]	333[b]
σ =	1,600	11	0.0019	0.0010	0.0034	0.10	0.0012	0.0013	0.0025		3,000	21
D14R $\bar{X}$ =	37,800	261	2.3727	1.0685	2.2205	29.06	2.7259	1.0765	2.5321	18.24	47,200	325
σ =	700	5	0.0009	0.0008	0.0020	0.06	0.0015	0.0010	0.0016		1,700	12
D15R $\bar{X}$ =	15,500	107	2.2078	1.0506	2.1016	32.86	2.6001	1.0596	2.4538	21.72	43,400	299
σ =	300	2	0.0006	0.0005	0.0012	0.03	0.0010	0.0011	0.0024		1,500	10

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6

Table 11 - Predrying laboratory data for OD compacts containing 100 percent portland cement and cured for twenty-eight days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)
d11r	$\bar{X}$ =	184,900	1,275	2.8679	1.1469	2.5007	20.11	3.1334	1.1578	2.7064
	σ =	1,800	12	0.0006	0.0003	0.0008	0.02	0.0004	0.0011	0.0025
d12r	$\bar{X}$ =	124,000	855	2.7093	1.1193	2.4206	22.66	2.9969	1.1270	2.6591
	σ =	3,500	24	0.0007	0.0013	0.0025	0.07	0.0015	0.0018	0.0031
d13r	$\bar{X}$ =	73,500	507	2.5390	1.0912	2.3268	25.66	2.8555	1.0982	2.6002
	σ =	1,800	12	0.0010	0.0008	0.0021	0.06	0.0008	0.0013	0.0032
d14r	$\bar{X}$ =	36,600	252	2.3729	1.0682	2.2213	29.03	2.7241	1.0741	2.5362
	σ =	1,000	7	0.0007	0.0005	0.0012	0.04	0.0012	0.0008	0.0016
d15r	$\bar{X}$ =	15,800	109	2.2069	1.0508	2.1003	32.90	2.5985	1.0580	2.4561
	σ =	700	5	0.0016	0.0013	0.0024	0.07	0.0021	0.0014	0.0031

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation

Table 12 - Postdrying laboratory data for OD compacts containing 100 percent portland cement and cured for twenty-eight days (sample size = 8 unless otherwise noted)

Series and Statistic[a]	24 Hour OD Weight (g)	42 Hour OD Weight (g)	48 Hour OD Weight (g)	48 Hour OD Volume (cm^3)	48 Hour OD Density (g/cm^3)	Measured Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
d11r $\bar{X}$ =	3.0234	3.0159	3.0141	1.1538	2.6124	10.30	69,200[c]	477[c]
σ =	0.0015	0.0013	0.0011	0.0012	0.0030		800	5
d12r $\bar{X}$ =	2.8660	2.8609	2.8606	1.1227	2.5479	12.09	61,400[b]	424[b]
σ =	0.0015	0.0013	0.0013	0.0014	0.0024		2,700	18
d13r $\bar{X}$ =	2.6980	2.6908	2.6909	1.0921	2.4638	14.99	57,200[b]	394[b]
σ =	0.0012	0.0014	0.0012	0.0007	0.0014		2,800	19
d14r $\bar{X}$ =	2.5308	2.5260	2.5255	1.0649	2.3717	18.49	54,000[c]	372[c]
σ =	0.0008	0.0008	0.0006	0.0010	0.0021		1,900	13
d15r $\bar{X}$ =	2.3720	2.3685	2.3690	1.0461	2.2646	21.69	48,700	336
σ =	0.0016	0.0015	0.0016	0.0011	0.0029		1,500	10

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7

Table 13 - Laboratory data for SSD compacts containing 100 percent portland cement and cured for ninety days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)	Calc. Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
E11	$\bar{X}$ =	192,000	1,323	2.8666	1.1513	2.4899	20.45	3.1127	1.1572	2.6898	10.25		
	σ =	3,000	21	0.0009	0.0017	0.0039	0.11	0.0029	0.0015	0.0022			
E12	$\bar{X}$ =	125,500[c]	866[c]	2.7063[c]	1.1177[c]	2.4214[c]	22.64[c]	2.9746[c]	1.1227[c]	2.6495[c]	12.09		
	σ =	6,100	42	0.0012	0.0012	0.0024	0.07	0.0028	0.0009	0.0031			
E13	$\bar{X}$ =	68,800	474	2.5360	1.0906	2.3253	25.71	2.8288	1.0958	2.5815	14.67		
	σ =	3,900	27	0.0019	0.0016	0.0033	0.09	0.0060	0.0017	0.0030			
E14	$\bar{X}$ =	33,300	229	2.3701	1.0676	2.2200	29.07	2.7030	1.0745	2.5156	17.50		
	σ =	1,500	10	0.0004	0.0013	0.0026	0.07	0.0030	0.0018	0.0024			
E15	$\bar{X}$ =	13,500[b]	93[b]	2.2051[b]	1.0525[b]	2.0952[b]	33.06[b]	2.5861[b]	1.0580[b]	2.4443[b]	20.85		
	σ =	900	6	0.0008	0.0025	0.0045	0.12	0.0026	0.0021	0.0030			

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 7
c. sample size = 9
Compressive strength data not available

Table 14 - Predrying laboratory data for OD compacts containing 100 percent portland cement and cured for ninety days (sample size = 9 unless otherwise noted)

Series and Statistic[a]		Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)
e11	$\bar{X}$ =	179,900	1,240	2.8667	1.1510	2.4906	20.43	3.1133	1.1579	2.6887
	σ =	8,200	56	0.0008	0.0032	0.0072	0.21	0.0038	0.0029	0.0044
e12	$\bar{X}$ =	127,700	880	2.7075	1.1191	2.4193	22.71	2.9787	1.1243	2.6495
	σ =	3,500	24	0.0007	0.0018	0.0036	0.10	0.0032	0.0027	0.0055
e13	$\bar{X}$ =	72,700	501	2.5368	1.0914	2.3244	25.74	2.8323	1.0967	2.5826
	σ =	2,300	16	0.0018	0.0013	0.0024	0.07	0.0051	0.0010	0.0033
e14	$\bar{X}$ =	33,700[b]	232[b]	2.3705[b]	1.0689[b]	2.2176[b]	29.15[b]	2.7062[b]	1.0739[b]	2.5199[b]
	σ =	1,400	10	0.0014	0.0019	0.0040	0.11	0.0039	0.0024	0.0039
e15	$\bar{X}$ =	14,700	102	2.2046	1.0503	2.0990	32.94	2.5834	1.0559	2.4466
	σ =	400	3	0.0015	0.0013	0.0035	0.10	0.0023	0.0011	0.0030

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 8

Table 15 - Postdrying laboratory data for OD compacts containing 100 percent portland cement and cured for ninety days (sample size = 9 unless otherwise noted)

Series and Statistic[a]		24 Hour OD Weight (g)	42 Hour OD Weight (g)	48 Hour OD Weight (g)	48 Hour OD Volume (cm^3)	48 Hour OD Density (g/cm^3)	Measured Final Porosity (percent)	Compressive Strength (psi) (MPa)
e11	$\bar{X}$ =	3.0091	2.9969	2.9949	1.1540	2.5952	10.23	
	σ =	0.0022	0.0024	0.0027	0.0029	0.0059		
e12	$\bar{X}$ =	2.8534	2.8439	2.8425	1.1191	2.5399	12.11	
	σ =	0.0032	0.0029	0.0030	0.0025	0.0052		
e13	$\bar{X}$ =	2.6775	2.6707	2.6696	1.0877	2.4543	14.84	
	σ =	0.0044	0.0050	0.0047	0.0013	0.0028		
e14	$\bar{X}$ =	2.5263[b]	2.5210[b]	2.5192[b]	1.0629[b]	2.3701[b]	17.41	
	σ =	0.0025	0.0026	0.0026	0.0019	0.0033		
e15	$\bar{X}$ =	2.3696	2.3641	2.3637	1.0427	2.2669	20.81	
	σ =	0.0023	0.0024	0.0026	0.0016	0.0047		

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 8
Compressive strength data not available

Table 16 - Laboratory data for SSD compacts containing ten percent sieved fly ash and cured for seven days (sample size = 8 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)	Calc. Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
C21S $\bar{X}$ =	176,300	1,215	2.8310	1.1534	2.4546	20.48	3.1055	1.1653	2.6649	11.72	45,100[b]	311[b]
σ =	1,900	13	0.0007	0.0019	0.0041	0.12	0.0019	0.0019	0.0030		700	5
C23S $\bar{X}$ =	63,800[d]	440[d]	2.5077[d]	1.0946[d]	2.2909[d]	25.78[d]	2.8286[d]	1.1030[d]	2.5644[d]	16.51	44,600[c]	308[c]
σ =	700	5	0.0026	0.0012	0.0020	0.06	0.0027	0.0023	0.0041		1,000	7
C25S $\bar{X}$ =	12,900	89	2.1801	1.0517	2.0729	32.84	2.5754	1.0624	2.4241	22.89	38,800[c]	267[c]
σ =	0	0	0.0005	0.0016	0.0032	0.09	0.0013	0.0010	0.0021		500	3

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7
d. sample size = 9

Table 17 - Predrying laboratory data for OD compacts containing ten percent sieved fly ash and cured for seven days (sample size = 8 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)
c21s $\bar{X}$ =	175,600	1,211	2.8320	1.1519	2.4587	20.35	3.1012	1.1602	2.6731
c21s σ =	2,500	17	0.0012	0.0009	0.0026	0.08	0.0011	0.0015	0.0035
c23s $\bar{X}$ =	64,300	443	2.5090	1.0927	2.2963	25.61	2.8258	1.0982	2.5731
c23s σ =	1,700	11	0.0027	0.0014	0.0026	0.07	0.0030	0.0025	0.0043
c25s $\bar{X}$ =	13,500	93	2.1793	1.0513	2.0729	32.84	2.5690	1.0565	2.4316
c25s σ =	300	2	0.0006	0.0005	0.0010	0.03	0.0006	0.0008	0.0017

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation

Table 18 - Postdrying laboratory data for OD compacts containing ten percent sieved fly ash and cured for seven days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		24 Hour OD Weight (g)	42 Hour OD Weight (g)	48 Hour OD Weight (g)	48 Hour OD Volume (cm^3)	48 Hour OD Density (g/cm^3)	Measured Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
c21s	$\bar{X}$ =	2.9781	2.9690	2.9676	1.1560	2.5671	11.52	66,300[b]	457[b]
	σ =	0.0009	0.0006	0.0006	0.0012	0.0028		1,700	12
c23s	$\bar{X}$ =	2.6501	2.6438	2.6445	1.0903	2.4254	16.51	56,600[b]	390[b]
	σ =	0.0033	0.0030	0.0031	0.0023	0.0051		1,900	13
c25s	$\bar{X}$ =	2.3313	2.3282	2.3278	1.0443	2.2290	22.83	42,800[b]	295[b]
	σ =	0.0005	0.0006	0.0005	0.0009	0.0020		1,500	10

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6

Table 19 - Laboratory data for SSD compacts containing ten percent sieved fly ash and cured for twenty-eight days (sample size = 8 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)	Calc. Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
D21SR $\bar{X}$ =	175,500	1,210	2.8319	1.1508	2.4608	20.28	3.1023	1.1609	2.6722	11.18	49,300	340
σ =	2,800	19	0.0021	0.0005	0.0015	0.04	0.0009	0.0019	0.0045		1,200	8
D23SRR$\bar{X}$ =	63,500[d]	438[d]	2.5073[d]	1.0940[d]	2.2919[d]	25.75[d]	2.8319[d]	1.1025[d]	2.5686[d]	15.69	45,500[b]	314[b]
σ =	2,100	15	0.0038	0.0007	0.0036	0.10	0.0024	0.0023	0.0045		1,400	10
D25SR $\bar{X}$ =	14,600[c]	101[c]	2.1797[c]	1.0507[c]	2.0745[c]	32.79[c]	2.5739[c]	1.0589[c]	2.4308[c]	21.49	43,500[c]	300[c]
σ =	300	2	0.0011	0.0005	0.0013	0.04	0.0016	0.0010	0.0022		900	6

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7
d. sample size = 9

Table 20 - Predrying laboratory data for OD compacts containing ten percent sieved fly ash and cured for twenty-eight days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)
d21sr	$\bar{X}$ =	174,500[b]	1,203[b]	2.8320[b]	1.1528[b]	2.4566[b]	20.41[b]	3.1075[b]	1.1632[b]	2.6716[b]
	σ =	1,700	12	0.0025	0.0009	0.0032	0.09	0.0027	0.0015	0.0028
d23sr	$\bar{X}$ =	64,000	441	2.5083	1.0958	2.2891	25.84	2.8500	1.1113	2.5646
	σ =	1,500	10	0.0021	0.0021	0.0042	0.12	0.0037	0.0021	0.0030
d25sr	$\bar{X}$ =	14,400	99	2.1802	1.0504	2.0755	32.76	2.5770	1.0603	2.4304
	σ =	600	4	0.0009	0.0013	0.0028	0.08	0.0009	0.0004	0.0012

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 9

Table 21 - Postdrying laboratory data for OD compacts containing ten percent sieved fly ash and cured for twenty-eight days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		24 Hour OD Weight (g)	42 Hour OD Weight (g)	48 Hour OD Weight (g)	48 Hour OD Volume (cm^3)	48 Hour OD Density (g/cm^3)	Measured Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
d21sr	$\bar{X}$ =	2.9811[c]	2.9760[c]	2.9754[c]	1.1595[c]	2.5661[c]	11.36	64,500[b]	445[b]
	σ =	0.0034	0.0031	0.0031	0.0011	0.0024		1,700	11
d23sr	$\bar{X}$ =	2.6863	2.6774	2.6762	1.1047	2.4226	15.64	56,700[b]	390[b]
	σ =	0.0035	0.0035	0.0036	0.0022	0.0043		2,000	14
d25sr	$\bar{X}$ =	2.3494	2.3464	2.3488	1.0506	2.2357	21.52	47,800	329
	σ =	0.0019	0.0018	0.0016	0.0005	0.0020		1,400	10

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 9

Table 22 - Laboratory data for SSD compacts containing ten percent sieved fly ash and cured for ninety days (sample size = 9 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)	Calc. Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
E21S $\bar{X}$ =	183,500[e]	1,265[e]	2.8328[e]	1.1556[e]	2.4513[e]	20.59[e]	3.1077[e]	1.1632[e]	2.6716[e]	11.70	49,400[d]	341[d]
σ =	3,200	22	0.0008	0.0013	0.0028	0.08	0.0017	0.0016	0.0026		2,000	14
E22S $\bar{X}$ =	125,600	866	2.6762	1.1246	2.3796	22.91	2.9743	1.1320	2.6276	13.64	49,300[c]	340[c]
σ =	1,500	10	0.0010	0.0015	0.0028	0.08	0.0025	0.0018	0.0023		1,200	8
E23S $\bar{X}$ =	76,900	530	2.5090	1.0960	2.2893	25.84	2.8353	1.1031	2.5703	16.08	48,800[c]	336[c]
σ =	2,600	18	0.0015	0.0013	0.0018	0.05	0.0022	0.0010	0.0027		700	5
E24S $\bar{X}$ =	36,500	252	2.3439	1.0712	2.1881	29.11	2.7055	1.0780	2.5097	18.80	47,100[d]	325[d]
σ =	900	6	0.0004	0.0010	0.0021	0.06	0.0010	0.0008	0.0012		1,400	10
E25S $\bar{X}$ =	15,800	109	2.1800	1.0519	2.0724	32.86	2.5814	1.0603	2.4345	21.92	46,700[b]	322[b]
σ =	500	3	0.0006	0.0008	0.0015	0.04	0.0014	0.0006	0.0014		600	4

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7
d. sample size = 8
e. sample size = 10

Table 23 - Predrying laboratory data for OD compacts containing ten percent sieved fly ash and cured for ninety days (sample size = 10 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	(MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)
e21s $\bar{X}$ =	184,400	1,271	2.8332	1.1566	2.4497	20.64	3.1107	1.1644	2.6715
σ =	3,900	27	0.0005	0.0016	0.0034	0.10	0.0016	0.0016	0.0023
e22s $\bar{X}$ =	125,000[c]	862[c]	2.6776[c]	1.1256[c]	2.3789[c]	22.93[c]	2.9754[c]	1.1314[c]	2.6299[c]
σ =	3,900	27	0.0003	0.0014	0.0027	0.08	0.0015	0.0018	0.0031
e23s $\bar{X}$ =	75,400	520	2.5090	1.0957	2.2898	25.82	2.8359	1.1026	2.5719
σ =	2,200	15	0.0006	0.0009	0.0018	0.05	0.0013	0.0017	0.0030
e24s $\bar{X}$ =	36,500[b]	252[b]	2.3424[b]	1.0714[b]	2.1862[b]	29.17[b]	2.7045[b]	1.0786[b]	2.5074[b]
σ =	1,600	11	0.0017	0.0009	0.0020	0.06	0.0018	0.0010	0.0022
e25s $\bar{X}$ =	15,100	104	2.1803	1.0525	2.0715	32.89	2.5808	1.0580	2.4393
σ =	600	4	0.0006	0.0009	0.0020	0.06	0.0011	0.0007	0.0012

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 8
c. sample size = 9

Table 24 - Postdrying laboratory data for OD compacts containing ten percent sieved fly ash and cured for ninety days (sample size = 10 unless otherwise noted)

Series and Statistic[a]		24 Hour OD Weight (g)	42 Hour OD Weight (g)	48 Hour OD Weight (g)	48 Hour OD Volume (cm^3)	48 Hour OD Density (g/cm^3)	Measured Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
e21s	$\bar{X}$ =	2.9850	2.9777	2.9766	1.1606	2.5648	11.52	65,100[b]	449[b]
	σ =	0.0010	0.0009	0.0009	0.0018	0.0043		2,200	15
e22s	$\bar{X}$ =	2.8298[d]	2.8226[d]	2.821[d]	1.1269[d]	2.5034[d]	13.64	62,000[b]	427[b]
	σ =	0.0008	0.0007	0.0007	0.0016	0.0033		1,500	11
e23s	$\bar{X}$ =	2.6660	2.6573	2.6572	1.0948	2.4270	16.57	54,700[b]	377[b]
	σ =	0.0009	0.0007	0.0007	0.0014	0.0032		2,000	14
e24s	$\bar{X}$ =	2.5087[c]	2.5045[c]	2.5032[c]	1.0691[c]	2.3413[c]	18.66	49,100[b]	338[b]
	σ =	0.0024	0.0023	0.0024	0.0012	0.0035		2,400	17
e25s	$\bar{X}$ =	2.3553	2.3495	2.3494	1.0471	2.2436	21.87	48,100[c]	331[c]
	σ =	0.0008	0.0008	0.0009	0.0011	0.0023		1,700	12

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 8
d. sample size = 9

Table 25 - Laboratory data for SSD compacts containing twenty percent sieved fly ash and cured for seven days (sample size = 9 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	(MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)	Calc. Final Porosity (percent)	Compressive Strength (psi)	(MPa)
C31S $\bar{X}$ =	168,800[d]	1,164[d]	2.7956[d]	1.1580[d]	2.4143[d]	20.70[d]	3.0763[d]	1.1694[d]	2.6307[d]	13.13	42,900[b]	296[b]
σ =	3,900	27	0.0009	0.0010	0.0025	0.07	0.0019	0.0013	0.0028		500	3
C33S $\bar{X}$ =	61,300	422	2.4771	1.0976	2.2568	25.87	2.8043	1.1082	2.5306	17.45	40,600[c]	280[c]
σ =	2,500	17	0.0016	0.0006	0.0014	0.04	0.0018	0.0029	0.0058		900	6
C35S $\bar{X}$ =	12,900	89	2.1522	1.0516	2.0465	32.78	2.5464	1.0605	2.4011	23.21	36,200[c]	250[c]
σ =	0	0	0.0007	0.0004	0.0011	0.03	0.0011	0.0012	0.0032		600	4

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7
d. sample size = 8

Table 26 - Predrying laboratory data for OD compacts containing twenty percent sieved fly ash and cured for seven days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)
c31s	$\bar{X}$ =	169,600[b]	1,169[b]	2.7963[b]	1.1587[b]	2.4133[b]	20.74[b]	3.0792[b]	1.1684[b]	2.6354[b]
	σ =	3,100	21	0.0004	0.0011	0.0022	0.06	0.0016	0.0014	0.0019
c33s	$\bar{X}$ =	65,000	448	2.4775	1.0973	2.2578	25.84	2.8049	1.1056	2.5369
	σ =	2,900	20	0.0005	0.0011	0.0025	0.07	0.0027	0.0029	0.0068
c35s	$\bar{X}$ =	13,300	92	2.1517	1.0513	2.0468	32.77	2.5459	1.0601	2.4016
	σ =	300	2	0.0020	0.0005	0.0019	0.06	0.0019	0.0005	0.0009

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 7

Table 27 - Postdrying laboratory data for OD compacts containing twenty percent sieved fly ash and cured for seven days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		24 Hour OD Weight (g)	42 Hour OD Weight (g)	48 Hour OD Weight (g)	48 Hour OD Volume (cm^3)	48 Hour OD Density (g/cm^3)	Measured Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
c31s	$\bar{X}$ =	2.9356[c]	2.9291[c]	2.9287[c]	1.1622[c]	2.5199[c]	12.88	62,000[b]	428[b]
	σ =	0.0013	0.0009	0.0011	0.0013	0.0024		1,500	11
c33s	$\bar{X}$ =	2.6104	2.6073	2.6068	1.0981	2.3740	17.92	51,900[c]	358[c]
	σ =	0.0023	0.0025	0.0024	0.0030	0.0084		1,900	13
c35s	$\bar{X}$ =	2.3048	2.3012	2.3022	1.0482	2.1963	22.99	39,100[b]	270[b]
	σ =	0.0020	0.0019	0.0018	0.0005	0.0018		1,100	8

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7

Table 28 - Laboratory data for SSD compacts containing twenty percent sieved fly ash and cured for twenty-eight days (sample size = 8 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)	Calc. Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
D31SR $\bar{X}$ =	175,000	1,206	2.7967	1.1568	2.4175	20.60	3.0806	1.1694	2.6343	13.01	44,100[b]	304[b]
σ =	2,700	19	0.0007	0.0015	0.0030	0.09	0.0016	0.0016	0.0028		1,000	7
D33SR $\bar{X}$ =	61,800[d]	426[d]	2.4776[d]	1.0964[d]	2.2597[d]	25.78[d]	2.8084[d]	1.1091[d]	2.5322[d]	17.25	42,500[c]	293[c]
σ =	1,100	8	0.0010	0.0014	0.0032	0.09	0.0025	0.0041	0.0076		1,600	11
D35SR $\bar{X}$ =	12,900	89	2.1512	1.0500	2.0487	32.71	2.5505	1.0607	2.4046	22.93	40,600[b]	280[b]
σ =	0	0	0.0022	0.0006	0.0025	0.07	0.0021	0.0013	0.0036		300	2

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7
d. sample size = 9

Table 29 - Predrying laboratory data for OD compacts containing twenty percent sieved fly ash and cured for twenty-eight days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)
d31sr	$\bar{X}$ =	172,300	1,188	2.7949	1.1565	2.4167	20.62	3.0780	1.1681	2.6351
	σ =	3,600	24	0.0031	0.0017	0.0023	0.07	0.0035	0.0036	0.0062
d33sr	$\bar{X}$ =	63,000[b]	434[b]	2.4771[b]	1.0962[b]	2.2597[b]	25.78[b]	2.8074[b]	1.1110[b]	2.5269[b]
	σ =	1,900	13	0.0004	0.0009	0.0018	0.05	0.0014	0.0030	0.0067
d35sr	$\bar{X}$ =	13,400	93	2.1520	1.0512	2.0473	32.76	2.5511	1.0614	2.4035
	σ =	200	1	0.0005	0.0008	0.0015	0.04	0.0008	0.0013	0.0029

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 9

Table 30 - Postdrying laboratory data for OD compacts containing twenty percent sieved fly ash and cured for twenty-eight days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		24 Hour OD Weight (g)	42 Hour OD Weight (g)	48 Hour OD Weight (g)	48 Hour OD Volume (cm^3)	48 Hour OD Density (g/cm^3)	Measured Final Porosity (percent)	Compressive Strength (psi)	(MPa)
d31sr	$\bar{X}$ =	2.9364	2.9280	2.9269	1.1628	2.5172	12.94	63,100	435
	σ =	0.0028	0.0026	0.0028	0.0044	0.0099		1,300	9
d33sr	$\bar{X}$ =	2.6206[c]	2.6144[c]	2.6140[c]	1.1012[c]	2.3739[c]	17.41	53,300[b]	367[b]
	σ =	0.0016	0.0018	0.0016	0.0029	0.0075		1,900	14
d35sr	$\bar{X}$ =	2.3135	2.3089	2.3080	1.0483	2.2017	22.90	45,000[b]	310[b]
	σ =	0.0006	0.0006	0.0004	0.0012	0.0025		1,100	8

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 9

Table 31 - Laboratory data for SSD compacts containing twenty percent sieved fly ash and cured for ninety days (sample size = 9 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)	Calc. Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
E31S $\bar{X}$ =	181,300[e]	1,250[e]	2.7982[e]	1.1597[e]	2.4130[e]	20.75[e]	3.0841[e]	1.1678[e]	2.6410[e]	12.76	46,100[d]	318[d]
σ =	4,000	28	0.0004	0.0009	0.0018	0.05	0.0014	0.0027	0.0048		2,100	14
E32S $\bar{X}$ =	118,600	818	2.6437	1.1288	2.3420	23.08	2.9499	1.1365	2.5955	14.56	45,500[b]	314[b]
σ =	2,800	19	0.0014	0.0012	0.0022	0.06	0.0017	0.0017	0.0027		1,200	8
E33S $\bar{X}$ =	66,800	461	2.4778	1.0970	2.2588	25.81	2.8123	1.1088	2.5363	16.66	45,800[c]	316[c]
σ =	1,200	8	0.0005	0.0005	0.0009	0.03	0.0012	0.0030	0.0062		2,000	13
E34S $\bar{X}$ =	30,400	210	2.3154	1.0714	2.1611	29.02	2.6812	1.0799	2.4828	19.13	44,700[c]	308[c]
σ =	1,000	7	0.0005	0.0010	0.0018	0.05	0.0011	0.0014	0.0027		1,500	11
E35S $\bar{X}$ =	12,800[e]	89[e]	2.1525[e]	1.0519[e]	2.0462[e]	32.79[e]	2.5592[e]	1.0620[e]	2.4098[e]	22.04	41,500[c]	286[c]
σ =	200	2	0.0010	0.0005	0.0016	0.05	0.0009	0.0012	0.0030		1,200	8

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7
d. sample size = 8
e. sample size = 10

Table 32 - Predrying laboratory data for OD compacts containing twenty percent sieved fly ash and cured for ninety days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)
e31s	$\bar{X}$ =	179,500[c]	1,238[c]	2.7965[c]	1.1594[c]	2.4121[c]	20.78[c]	3.0833[c]	1.1688[c]	2.6380[c]
	σ =	2,800	19	0.0004	0.0006	0.0013	0.04	0.0021	0.0030	0.0051
e32s	$\bar{X}$ =	117,900[b]	813[b]	2.6430[b]	1.1287[b]	2.3416[b]	23.09[b]	2.9517[b]	1.1377[b]	2.5944[b]
	σ =	1,600	11	0.0007	0.0012	0.0021	0.06	0.0032	0.0017	0.0039
e33s	$\bar{X}$ =	67,800	467	2.4774	1.0971	2.2581	25.83	2.8129	1.1077	2.5395
	σ =	1,100	8	0.0006	0.0007	0.0015	0.04	0.0014	0.0025	0.0048
e34s	$\bar{X}$ =	31,300	216	2.3139	1.0713	2.1598	29.06	2.6799	1.0799	2.4816
	σ =	900	6	0.0017	0.0005	0.0019	0.06	0.0013	0.0013	0.0031
e35s	$\bar{X}$ =	12,900[c]	89[c]	2.1528[c]	1.0518[c]	2.0468[c]	32.77[c]	2.5599[c]	1.0613[c]	2.4120[c]
	σ =	0	0	0.0014	0.0005	0.0014	0.04	0.0013	0.0007	0.0012

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 9
c. sampl size = 10

Table 33 - Postdrying laboratory data for OD compacts containing twenty percent sieved fly ash and cured for ninety days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		24 Hour OD Weight (g)	42 Hour OD Weight (g)	48 Hour OD Weight (g)	48 Hour OD Volume (cm^3)	48 Hour OD Density (g/cm^3)	Measured Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
e31s	$\bar{X}$ =	2.9434[e]	2.9380[e]	2.9369[e]	1.1649[e]	2.5213[e]	12.53	57,900[c]	399[c]
	σ =	0.0014	0.0012	0.0012	0.0027	0.0065		1,200	8
e32s	$\bar{X}$ =	2.7928[d]	2.7861[d]	2.7849[d]	1.1322[d]	2.4597[d]	14.66	54,600[b]	377[b]
	σ =	0.0015	0.0015	0.0014	0.0015	0.0031		2,400	17
e33s	$\bar{X}$ =	2.6325	2.6243	2.6245	1.0989	2.3883	17.01	55,900[b]	386[b]
	σ =	0.0011	0.0010	0.0010	0.0022	0.0053		2,000	14
e34s	$\bar{X}$ =	2.4792	2.4740	2.4736	1.0705	2.3108	19.10	51,700	357
	σ =	0.0015	0.0013	0.0014	0.0006	0.0021		1,200	8
e35s	$\bar{X}$ =	2.3324[e]	2.3287[e]	2.3274[e]	1.0499[e]	2.2168[e]	21.91	44,200	304
	σ =	0.0016	0.0017	0.0017	0.0009	0.0018		2,000	14

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7
d. sample size = 9
e. sample size = 10

Table 34 - Laboratory data for SSD compacts containing thirty percent sieved fly ash and cured for seven days (sample size = 8 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)	Calc. Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
C41SR $\bar{X}$ =	166,000	1,145	2.7619	1.1611	2.3788	20.80	3.0529	1.1739	2.6006	14.89	37,100[b]	256[b]
σ =	4,300	29	0.0018	0.0012	0.0016	0.05	0.0024	0.0017	0.0029		1,100	8
C43SR $\bar{X}$ =	60,200[c]	415[c]	2.4472[c]	1.0974[c]	2.2301[c]	25.75[c]	2.7785[c]	1.1101[c]	2.5029[c]	18.57	38,200[c]	263[c]
σ =	2,300	16	0.0009	0.0010	0.0026	0.08	0.0006	0.0031	0.0069		1,600	11
C45SRR$\bar{X}$ =	12,600	87	2.1249	1.0530	2.0180	32.81	2.5208	1.0607	2.3764	23.82	32,400[c]	223[c]
σ =	400	3	0.0006	0.0008	0.0018	0.05	0.0009	0.0014	0.0035		1,500	11

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7

Table 35 - Predrying laboratory data for OD compacts containing thirty percent sieved fly ash and cured for seven days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)
c41s	$\bar{X}$ =	170,100[b]	1,173[b]	2.7608[b]	1.1634[b]	2.3730[b]	20.99[b]	3.0511[b]	1.1753[b]	2.5962[b]
	σ =	3,200	22	0.0019	0.0008	0.0027	0.08	0.0013	0.0022	0.0045
c43sr	$\bar{X}$ =	61,400	424	2.4451	1.0978	2.2273	25.85	2.7779	1.1080	2.5071
	σ =	800	6	0.0017	0.0013	0.0032	0.09	0.0019	0.0023	0.0047
c45sr	$\bar{X}$ =	12,700[c]	88[c]	2.1248[c]	1.0512[c]	2.0213[c]	32.71[c]	2.5195[c]	1.0598[c]	2.3774[c]
	σ =	300	2	0.0022	0.0005	0.0017	0.05	0.0018	0.0016	0.0027

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 7
c. sample size = 9

Table 36 - Postdrying laboratory data for OD compacts containing thirty percent sieved fly ash and cured for seven days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		24 Hour OD Weight (g)	42 Hour OD Weight (g)	48 Hour OD Weight (g)	48 Hour OD Volume (cm^3)	48 Hour OD Density (g/cm^3)	Measured Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
c41s	$\bar{X}$ =	2.8784[c]	2.8748[c]	2.8746[c]	1.1682[c]	2.4608[c]	15.02	55,700[b]	384[b]
	σ =	0.0028	0.0026	0.0026	0.0029	0.0080		900	6
c43sr	$\bar{X}$ =	2.5732	2.5711	2.5712	1.0997	2.3381	18.66	47,400[b]	327[b]
	σ =	0.0025	0.0022	0.0022	0.0026	0.0068		2,300	16
c45sr	$\bar{X}$ =	2.2720[d]	2.2680[d]	2.2679[d]	1.0480[d]	2.1641[d]	23.74	37,900[b]	261[b]
	σ =	0.0020	0.0020	0.0021	0.0011	0.0028		1,700	12

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 7
d. sample size = 9

Table 37 - Laboratory data for SSD compacts containing thirty percent sieved fly ash and cured for twenty-eight days (sample size = 7 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)	Calc. Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
D41SR $\bar{X}$ =	171,900[d]	1,185[d]	2.7602[d]	1.1610[d]	2.3774[d]	20.85[d]	3.0529[d]	1.1739[d]	2.6006[d]	14.13	40,700	281
σ =	2,000	14	0.0006	0.0010	0.0021	0.06	0.0017	0.0018	0.0035		900	6
D43SR $\bar{X}$ =	56,300	388	2.4460	1.0979	2.2280	25.82	2.7828	1.1131	2.5001	17.94	42,000[b]	289[b]
σ =	800	6	0.0006	0.0015	0.0033	0.10	0.0022	0.0039	0.0072		1,800	12
D45SR $\bar{X}$ =	12,800[c]	88[c]	2.1258[c]	1.0510[c]	2.0227[c]	32.66[c]	2.5290[c]	1.0624[c]	2.3804[c]	23.18	38,400	265
σ =	200	2	0.0005	0.0003	0.0008	0.02	0.0011	0.0008	0.0022		700	5

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 6
c. sample size = 8
d. sample size = 9

Table 38 - Predrying laboratory data for OD compacts containing thirty percent sieved fly ash and cured for twenty-eight days (sample size = 6 unless otherwise noted)

Series and Statistic[a]		Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)
d41sr	$\bar{X}$ =	169,000	1,165	2.7590	1.1592	2.3800	20.76	3.0546	1.1757	2.5982
	σ =	2,300	16	0.0006	0.0011	0.0024	0.07	0.0016	0.0029	0.0055
d43sr	$\bar{X}$ =	56,300	388	2.4464	1.0988	2.2263	25.88	2.7846	1.1166	2.4938
	σ =	1,100	7	0.0005	0.0008	0.0016	0.05	0.0025	0.0043	0.0082
d45sr	$\bar{X}$ =	12,300[b]	85[b]	2.1232[b]	1.0505[b]	2.0212[b]	32.71[b]	2.5278[b]	1.0656[b]	2.3723[b]
	σ =	200	2	0.0008	0.0010	0.0020	0.06	0.0017	0.0014	0.0032

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 8

Table 39 - Postdrying laboratory data for OD compacts containing thirty percent sieved fly ash and cured for twenty-eight days (sample size = 6 unless otherwise noted)

Series and Statistic[a]	24 Hour OD Weight (g)	42 Hour OD Weight (g)	48 Hour OD Weight (g)	48 Hour OD Volume (cm^3)	48 Hour OD Density (g/cm^3)	Measured Final Porosity (percent)	Compressive Strength (psi)	(MPa)
d41sr $\bar{X}$ =	2.9331	2.8972	2.8916	1.1690	2.4736	13.86	54,200	374
σ =	0.0018	0.0013	0.0010	0.0025	0.0057		1,800	12
d43sr $\bar{X}$ =	2.2587	2.5800	2.5798	1.1080	2.3284	18.34	50,100	346
σ =	0.0018	0.0015	0.0016	0.0050	0.0115		1,500	10
d45sr $\bar{X}$ =	2.2992[b]	2.2840[b]	2.2820[b]	1.0517[b]	2.1698[b]	23.07	41,400	285
σ =	0.0017	0.0017	0.0017	0.0013	0.0026		800	5

Note:
a. X = sample average and o = sample standard deviation
b. sample size = 8

Table 40 - Laboratory data for SSD compacts containing thirty percent sieved fly ash and cured for ninety days (sample size = 9 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)	Calc. Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
E41S $\bar{X}$ =	175,500	1,210	2.7614	1.1634	2.3735	20.98	3.0594	1.1748	2.6041	14.41	43,400[b]	299[b]
σ =	2,800	19	0.0011	0.0009	0.0019	0.06	0.0022	0.0032	0.0056		1,300	9
E42S $\bar{X}$ =	116,200[c]	801[c]	2.6083[c]	1.1310[c]	2.3062[c]	23.22[c]	2.9234[c]	1.1413[c]	2.5614[c]	16.13	42,900[b]	296[b]
σ =	3,100	21	0.0010	0.0013	0.0024	0.07	0.0022	0.0019	0.0029		1,000	7
E43S $\bar{X}$ =	68,700	474	2.4468	1.0996	2.2251	25.92	2.7896	1.1118	2.5091	18.21	43,300[b]	299[b]
σ =	1,600	11	0.0005	0.0009	0.0018	0.05	0.0009	0.0021	0.0040		1,000	7
E44S $\bar{X}$ =	31,200[c]	215[c]	2.2844[c]	1.0718[c]	2.1313[c]	29.04[c]	2.6565[c]	1.0808[c]	2.4580[c]	20.61	42,400[b]	292[b]
σ =	700	5	0.0009	0.0005	0.0012	0.03	0.0038	0.0006	0.0037		1,100	7
E45S $\bar{X}$ =	12,600[d]	87[d]	2.1240[d]	1.0516[d]	2.0198[d]	32.75[d]	2.5327[d]	1.0603[d]	2.3886[d]	23.46	40,200	277
σ =	500	3	0.0014	0.0006	0.0019	0.06	0.0015	0.0008	0.0019		1,600	11

Note:
a. $\bar{X}$ = sample average and σ sample standard deviation
b. sample size = 6
c. sample size = 8
d. sample size = 10

Table 41 - Predrying laboratory data for OD compacts containing thirty percent sieved fly ash and cured for ninety days (sample size = 8 unless otherwise noted)

Series and Statistic[a]	Production Pressure (psi)	Production Pressure (MPa)	Initial Weight (g)	Initial Volume (cm^3)	Initial Density (g/cm^3)	Calc. Initial Porosity (percent)	SSD Weight (g)	SSD Volume (cm^3)	SSD Density (g/cm^3)
e41s $\bar{X}$ =	174,000	1,200	2.7599	1.1633	2.3725	21.01	3.0585	1.1741	2.6050
e41s σ =	3,200	22	0.0018	0.0010	0.0019	0.06	0.0024	0.0024	0.0045
e42s $\bar{X}$ =	112,100[b]	773[b]	2.6079[b]	1.1313[b]	2.3053[b]	23.25[b]	2.9241[b]	1.1437[b]	2.5567[b]
e42s σ =	2,100	14	0.0014	0.0011	0.0019	0.06	0.0024	0.0024	0.0037
e43s $\bar{X}$ =	66,700	460	2.4451	1.0996	2.2236	25.97	2.7872	1.1125	2.5054
e43s σ =	2,000	14	0.0017	0.0011	0.0029	0.09	0.0022	0.0032	0.0062
e44s $\bar{X}$ =	31,000[c]	214[c]	2.2841[c]	1.0726[c]	2.1296[c]	29.10[c]	2.6553[c]	1.0812[c]	2.4559[c]
e44s σ =	1,300	9	0.0013	0.0013	0.0030	0.09	0.0025	0.0022	0.0031
e45s $\bar{X}$ =	12,500[b]	86[b]	2.1237[b]	1.0510[b]	2.0207[b]	32.72[b]	2.5320[b]	1.0592[b]	2.3906[b]
e45s σ =	500	3	0.0007	0.0007	0.0014	0.04	0.0018	0.0012	0.0016

Note:
a. $\bar{X}$ = sample average and σ = sample standard deviation
b. sample size = 9
c. sample size = 10

Table 42 - Postdrying laboratory data for OD compacts containing thirty percent sieved fly ash and cured for ninety days (sample size = 8 unless otherwise noted)

Series and Statistic[a]		24 Hour OD Weight (g)	42 Hour OD Weight (g)	48 Hour OD Weight (g)	48 Hour OD Volume (cm^3)	48 Hour OD Density (g/cm^3)	Measured Final Porosity (percent)	Compressive Strength (psi)	Compressive Strength (MPa)
e41s	$\bar{X}$ =	2.8901	2.8891	2.8891	1.1680	2.4736	14.43	56,100[b]	386[b]
	σ =	0.0023	0.0022	0.0020	0.0026	0.0055		900	6
e42s	$\bar{X}$ =	2.7405[d]	2.7357[d]	2.7355[d]	1.1346[d]	2.4110[d]	16.49	54,900[c]	379[c]
	σ =	0.0016	0.0017	0.0015	0.0018	0.0033		900	6
e43s	$\bar{X}$ =	2.5956	2.5877	2.5868	1.1017	2.3481	18.01	49,100[b]	338[b]
	σ =	0.0027	0.0023	0.0023	0.0021	0.0056		1,300	9
e44s	$\bar{X}$ =	2.4367[e]	2.4340[e]	2.4355[e]	1.0702[e]	2.2758[e]	20.33	46,800	323
	σ =	0.0015	0.0018	0.0017	0.0020	0.0043		700	5
e45s	$\bar{X}$ =	2.2835[d]	2.2793[d]	2.2806[d]	1.0470[d]	2.1783[d]	23.73	45,200[b]	311[b]
	σ =	0.0017	0.0021	0.0016	0.0012	0.0023		900	6

Note:
a. $\bar{X}$ = sample average and σ sample standard deviation
b. sample size = 6
c. sample size = 7
d. sample size = 9
e. sample size = 10

APPENDIX F

LINEAR REGRESSION SUMMARY

Introduction

The purpose of this Appendix is to provide additional pertinent data concerning the various linear regression analyses performed for this report. The data presented will be sorted by the figure which contains the particular linear relationship in question. Additional data presented includes the units to be used for the X and Y axis values, the slope of the developed regression equation, the Y-intercept of the regression line, the number of data points used for the equation development or degrees of freedom and the coefficient of determination for each equation.

Table 43 - Linear regression summary data

Figure Number	Line Description	Axis Units		Degrees of Freedom	Slope of Regression Line	Y-Intercept of Regression Line	Coefficient of Determination
		Y	X				
10	0% Fly Ash	grams	percent	40	-0.051	3.887	0.993
	10% Fly Ash	grams	percent	22	-0.052	3.887	0.994
	20% Fly Ash	grams	percent	22	-0.053	3.877	0.993
	30% Fly Ash	grams	percent	22	-0.053	3.857	0.992
11	0% Fly Ash	grams	$\log_{10}$ X, psi	40	0.59	-0.28	0.978
	10% Fly Ash	grams	$\log_{10}$ X, psi	22	0.58	-0.27	0.976
	20% Fly Ash	grams	$\log_{10}$ X, psi	22	0.56	-0.18	0.983
	30% Fly Ash	grams	$\log_{10}$ X, psi	22	0.55	-0.16	0.983
12		percent	$\log_{10}$ X, psi	106	-10.9	78.2	0.991
13	0% Fly Ash	percent	$\log_{10}$ X, psi	20	-10.5	65.9	0.979
	10% Fly Ash	percent	$\log_{10}$ X, psi	11	-9.5	61.9	0.983
	20% Fly Ash	percent	$\log_{10}$ X, psi	11	-8.6	58.1	0.980
	30% Fly Ash	percent	$\log_{10}$ X, psi	11	-7.9	55.7	0.987
14	SSD	g/cm^3	$\log_{10}$ X, psi	40	0.22	1.5	0.991
	OD	g/cm^3	$\log_{10}$ X, psi	40	0.32	0.9	0.994
	Initial	g/cm^3	$\log_{10}$ X, psi	20	0.36	0.6	0.994
15	SSD	g/cm^3	$\log_{10}$ X, psi	22	0.22	1.5	0.992
	OD	g/cm^3	$\log_{10}$ X, psi	11	0.30	1.0	0.995
	Initial	g/cm^3	$\log_{10}$ X, psi	22	0.35	0.6	0.991
16	SSD	g/cm^3	$\log_{10}$ X, psi	22	0.20	1.6	0.995
	OD	g/cm^3	$\log_{10}$ X, psi	11	0.28	1.1	0.992
	Initial	g/cm^3	$\log_{10}$ X, psi	22	0.32	0.7	0.997

Table 43 continued - Linear regression summary data

Figure Number	Line Description	Axis Units		Degrees of Freedom	Slope of Regression Line	Y-Intercept of Regression Line	Coefficient of Determination
		Y	X				
17	SSD	g/cm^3	$\log_{10}$ X, psi	22	0.19	1.6	0.994
	OD	g/cm^3	$\log_{10}$ X, psi	11	0.26	1.1	0.995
	Initial	g/cm^3	$\log_{10}$ X, psi	22	0.31	0.7	0.996
18	Initial	percent	$\log_{10}$ X, psi	106	-10.93	78.2	0.991
	Final	percent	$\log_{10}$ X, psi	48	-9.18	60.5	0.919
19	14,000 psi	percent	percent	12	-0.19	34.4	0.679
	70,000 psi	percent	percent	12	-0.39	41.7	0.918
	175,000 psi	percent	percent	12	-0.57	48.4	0.944
20	3 day	psi	$\log_{10}$ X, psi	5	7,731.7	2,336	0.929
	7 day	psi	$\log_{10}$ X, psi	5	6,053.3	16,870	0.933
	28 day	psi	$\log_{10}$ X, psi	5	5,691.2	20,291	0.912
21	7 day	psi	$\log_{10}$ X, psi	3	5,797.9	15,464	0.898
	28 day	psi	$\log_{10}$ X, psi	3	5,211.7	21,412	0.922
	90 day	psi	$\log_{10}$ X, psi	5	2,877.0	34,445	0.936
22	7 day	psi	$\log_{10}$ X, psi	3	6,041.7	11,453	0.997
	28 day	psi	$\log_{10}$ X, psi	3	3,066.9	27,940	0.996
	90 day	psi	$\log_{10}$ X, psi	5	3,643.6	27,415	0.807
23	7 day	psi	$\log_{10}$ X, psi	3	4,566.1	14,439	0.699
	28 day	psi	$\log_{10}$ X, psi	3	2,227.7	29,902	0.478
	90 day	psi	$\log_{10}$ X, psi	5	2,575.1	30,213	0.807
24	7 to 28 days	percent	$\log_{10}$ X, psi	11	-1.3	10.1	0.996

Table 43 continued - Linear regression summary data

Figure Number	Line Description	Axis Units		Degrees of Freedom	Slope of Regression Line	Y-Intercept of Regression Line	Coefficient of Determination
		Y	X				
25	7 to 28 days	percent	$\log_{10}$ X, psi	11	-2.4	19.0	0.996
	7 to 90 days	percent	$\log_{10}$ X, psi	11	-8.7	53.4	0.996
26	7 to 28 days	percent	$\log_{10}$ X, psi	11	-8.6	47.1	0.996
	7 to 90 days	percent	$\log_{10}$ X, psi	11	-8.0	49.6	0.996
27	7 to 28 days	percent	$\log_{10}$ X, psi	11	-8.0	55.5	0.996
	7 to 90 days	percent	$\log_{10}$ X, psi	11	-8.3	51.5	0.996
28	0% Fly Ash,SSD	psi	percent	5	-621.5	55,539	0.946
	10% Fly Ash,SSD	psi	percent	3	-582.6	52,760	0.869
	20% Fly Ash,SSD	psi	percent	3	-670.0	51,913	0.991
	30% Fly Ash,SSD	psi	percent	3	-565.8	46,702	0.679
	0% Fly Ash,OD	psi	percent	5	-1868.7	87,399	0.957
	10% Fly Ash,OD	psi	percent	3	-2082.4	90,537	0.997
	20% Fly Ash,OD	psi	percent	3	-2265.3	91,618	0.996
	30% Fly Ash,OD	psi	percent	3	-2030.4	85,862	0.997
29	0% Fly Ash,SSD	psi	percent	5	-512.9	55,555	0.866
	10% Fly Ash,SSD	psi	percent	3	-552.4	55,004	0.939
	20% Fly Ash,SSD	psi	percent	3	-351.8	48,637	0.999
	30% Fly Ash,SSD	psi	percent	3	-280.3	45,529	0.488
	0% Fly Ash,OD	psi	percent	5	-1612.3	83,111	0.933
	10% Fly Ash,OD	psi	percent	3	-1635.9	82,791	0.997
	20% Fly Ash,OD	psi	percent	3	-1805.8	85,853	0.989
	30% Fly Ash,OD	psi	percent	3	-1394.0	74,248	0.965

Table 43 continued - Linear regression summary data

Figure Number	Line Description	Axis Units		Degrees of Freedom	Slope of Regression Line	Y-Intercept of Regression Line	Coefficient of Determination
		Y	X				
30	10% Fly Ash,SSD	psi	percent	5	-299.8	53,185	0.920
	20% Fly Ash,SSD	psi	percent	5	-451.9	52,416	0.784
	30% Fly Ash,SSD	psi	percent	5	-319.9	48,378	0.764
	10% Fly Ash,OD	psi	percent	5	-1804.3	85,485	0.940
	20% Fly Ash,OD	psi	percent	5	-1325.4	75,447	0.831
	30% Fly Ash,OD	psi	percent	5	-1269.9	74,038	0.880
31	7 day	percent	percent	24	-0.7	0.6	0.988
	28 day	percent	percent	24	-0.5	4.7	0.869
	90 day	percent	percent	18	-0.6	11.6	0.758

VITA

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Jeff Bormann was born on September 14, 1958, the son of Grace and Elroy Bormann. Growing up in the small farming community of Geronimo, Texas, he attended New Braunfels Canyon High School where he graduated in May, 1977.

In August, 1977 Jeff enrolled in Texas A&M University where he recieved a Bachelor of Science degree in Civil Engineering, Cum Laude, in December, 1981. During his undergraduate career, Jeff was a member of the student chapter of the ASCE, the Chi Epsilon National Civil Engineering Honor Society, the Tau Beta Pi National Engineering Honor Society, a recipient of the Dahlstrom Corporation Scholarship in Civil Engineering, a letterman on the 1978 SWC Champion Texas Aggie Baseball team and a Davis-Gary Fighter.

Upon graduation in 1981, Jeff was employed by the H.B. Zachry Company of San Antonio, Texas as a Planning and Scheduling Engineer. He was assigned to the Conoco HDPE plant in Bay City, Texas from January, 1982 to August, 1983.

In August, 1983 Jeff enrolled in the Masters Program in Construction Management at Texas A&M University. As a graduate research assistant, he became involved with a project to develop ultra-high strength concrete sponsored by the U.S. Air Force. Jeff is expected to receive his Master of Science in Civil Engineering in August, 1985 and will return to the H.B. Zachry Company.